

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicant(s) : Awdhoot Vasant Kerkar, et al.  
Serial No : 10/812,541  
Filed : March 30, 2004  
For : ROOFING SHINGLES PROVIDED WITH  
RELEASE COATING  
Examiner : Alicia Ann Chevalier  
Art Unit : 1794  
Docket : FDN 2831 (040001U034)  
Dated : October 14, 2008

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**APPEAL BRIEF**

This appeal brief is in response to the Final Office Action dated July 25, 2007 and the Advisory Action dated November 20, 2007, received in connection with the above-captioned application. Appellants respectfully request reconsideration and reversal of the rejections of those Office Actions for the reasons stated bellow.

Please charge the fee due under 37 C.F.R. §41.20(b)(2) to Deposit Account No. 03-1250.

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(1) REAL PARTY IN INTEREST

Building Materials Investment Corporation ("BMIC") is the real party in interest, appellants having assigned to BMIC the entire right, title, and interest in and to, *inter alia*, the present application.

(2) RELATED APPEALS AND INTERFERENCES

Appellants, Appellants' assignee, and the undersigned are aware of no related appeals or interferences that may be related to, directly affect or be directly affected by, or have a bearing on the Board's decision in the pending appeal.

(3) STATUS OF CLAIMS

The pending claims are 1-3, 5-8 and 10-18. No claims have been allowed.

Claims 4 and 9 have been canceled.

Claims 11-18 have been withdrawn from consideration but not canceled.

Claims 1-3, 5-8 and 10 stand rejected; the rejection of these pending claims is appealed herein.

A copy of the appealed claims is contained in Item (8) of this Appeal, the Claims Appendix (beginning on page 16 of this paper).

(4) STATUS OF AMENDMENTS

An Amendment and Response to the Final Office Action was filed, and the amendments contained therein were entered and considered by the Examiner. All amendments have been entered.

(5) SUMMARY OF CLAIMED SUBJECT MATTER

The claims on appeal relate to roofing shingles provided with a release coating, and more specifically, to roofing shingles of which a pressure point portion of the bottom surface is provided with a release coating of a continuous film of particles.

Roofing shingles typically comprise a top ("anterior") and bottom ("posterior") layer, and are often stacked in piles when packaged, such that the anterior layer of a first shingle faces the posterior layer of an adjoining shingle. However, the high pressure caused by the stacking of shingles can cause the shingles to stick together. For many types of shingles, this is especially pronounced at a pressure point resulting from a sharp transition in thickness between the unbonded top portion of the anterior layer and the portion of that layer bonded to the posterior layer. This pressure point results in distortion and localized sticking of the shingles.

The presently claimed invention addresses these problems by providing improved shingles that are coated at a pressure point portion of the bottom surface with a release coating containing particles that result in a film with poor interlaminar strength and good to perfect basal cleavage. These particles peel away from each other or are easily fractured, significantly reducing the force required to separate adjacent shingles from each other, and enabling easy separation.

The following is a summary of the currently appealed claims:

1. A roofing shingle comprising a top and bottom surface {**Figure 1 and Figure 2; page 4, lines 20-21**}, the bottom surface provided with a release coating of a continuous film of particles having good to perfect basal cleavage {**page 5, lines 13-30**}, wherein

said release coating is disposed only on a pressure point portion of said bottom surface {page 9, lines 15-20}, wherein said particles are in the class phyllosilicates {page 5, lines 13-19}.

2. A shingle in accordance with claim 1 wherein said pressure point portion is immediately above and below a top edge of said bottom surface where the top edge overlaps a bottom portion of a headlap portion of said top surface {Figure 2; page 9, lines 13-19}.

3. A shingle in accordance with claim 1 wherein said roofing shingle is a shingle selected from the group consisting of a laminated shingle and a strip shingle {page 1, lines 18-19}.

5. A shingle in accordance with claim 1 wherein said particles are selected from the group consisting of Allophane, Apophyllite, Bannisterite, Carletonite, Cavansite, Chrysocolla, Baileychlore, Chamosite, Chlorite, Clinochlore, Cookeite, Nimitite, Pennantite, Penninite, Sudioite, Glauconite, Illite, Kaolinite, Montmorillonite, Palygorskite, Pyrophyllite, Sauconite, Talc, Vermiculite, Delhayelite, Elpidite, Fedorite, Franklinfumaceite, Franklinphilite, Gonyerite, Gyolite and Leucosphenite; Biotite, Lepidolite, Muscovite, Paragonite, Phlogopite, Zinnwaldite, Minehillite, Nordite, Pentagonite, Petalite, Prehnite, Rhodesite, Sanfomite, Antigorate, Clinochrysotile, Lizardite, Orthochrysotile, Serpentine, Wickenburgite, Zeophyllite {page 6, line 6 – page 9, line 5}.



6. A stack of roofing shingles comprising a plurality of said roofing shingles of claim 1 wherein the top surface of each roofing shingle faces the bottom surface of its adjoining roofing shingle {page 1, line 30 – page 2, line 20} .
7. A stack of roofing shingles comprising a plurality of said roofing shingles of claim 2 wherein the top surface of each roofing shingle faces the bottom surface of its adjoining roofing shingle {page 9, lines 22-25}.
8. A stack of roofing shingles comprising a plurality of said roofing shingles of claim 3 wherein the top surface of each roofing shingle faces the bottom surface of its adjoining roofing shingle {page 9, lines 22-25}.
10. A stack of roofing shingles comprising a plurality of said roofing shingles of claim 5 wherein the top surface of each roofing shingle faces the bottom surface of its adjoining roofing shingle{page 9, lines 22-25}.

(6) GROUND OF REJECTION TO BE REVIEWED ON APPEAL

Whether 1-3, 5-8 and 10 are obvious under 35 U.S.C. § 103(a) based on U.S. Patent No. 4,738,884 to Algrim et al. (“Algrim”)<sup>1</sup> in view of U.S. Patent No. 2,326,724 to Fasold et al. (“Fasold”)<sup>2</sup> and evidenced by the Wikipedia entry for “Basal Cleavage” printed on January 7, 2007 (“Wikipedia”)<sup>3</sup>.

No other rejections are pending.

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<sup>1</sup> Algrim is attached as **Exhibit A**.

<sup>2</sup> Fasold is attached as **Exhibit B**.

<sup>3</sup> Wikipedia is attached as **Exhibit C**.

(7) ARGUMENT

Appellants respectfully request reversal of the rejections of the Final Office Action and Advisory Action, submitting that the present invention is not obvious in view of the combination of Algrim and Fasold, as evidenced by Wikipedia.

**A. Claims 1-3, 5-8 and 10 Rejected Under 35 U.S.C. § 103(a)**

Responding to the rejection of claims 1-3, 5-8 and 10 under 35 USC § 103(a), the Appellants respectfully request reconsideration based on the non-obviousness of the present claims in view of the Algrim and Fasold, as evidenced by Wikipedia.

Specifically, it has been alleged that Algrim discloses a roofing shingle having a bottom surface with a release coating of a continuous film where the release coating is disposed only on a pressure point portion of the bottom surface. It is acknowledged that Algrim fails to disclose that the particles have good to perfect basal cleavage, but it is alleged that Fasold teaches the presence of talc, and that the particles on the bottom surface prevent sticking of the adjacent layers of the roofing material in a package (as evidenced by Wikipedia, which teaches that “basal cleavage” refers to cleavage parallel to the base of a crystal, or to the plane of the lateral axes, making the material feel slippery). It appears that the Examiner is arguing the following:

- (a) If Algrim teaches application of silicone or fluorocarbons to a paper or polyester release material at the “pressure point” to prevent adhesion; and
- (b) If Fasold teaches application of talc to the entire surface to prevent adhesion;

(c) Then the combination of Algrim and Fasold (evidenced by Wikipedia) teaches the application of phyllosilicate particles to the “pressure point” to optimize the prevention of adhesion.

However, as stated previously and reiterated here, such a conclusion is not supported by the art cited.

The present invention provides for a release coating comprising phyllosilicate particles applied on a pressure point portion of a bottom surface of the shingle. As taught in the Specification, optimal results, in terms of application and material costs, are obtained when the coating is applied to the “pressure point” portion of the laminate shingle. This pressure point is generally denoted by reference numeral 6 in FIG. 2. Pressure is greatest in the stack at this location. As such, it is at this point where adhesion between shingles is most apt to occur. Thus, this region is where application of a release coating comprising a continuous film of phyllosilicate particles, to prevent adhesion, is most preferred. This solution is advantageous over those known in the art because Appellants have discovered that phyllosilicates are especially useful for preventing adhesion, and by providing such particles directly to the shingle, along the “pressure point” of the shingle’s surface, optimal prevention of adhesion can be achieved with minimal cost and disruption to the shingle design and manufacturing process.

Algrim teaches that the release material 14 is *paper or polyester which “have to be treated with a non-adhering substance such as silicone or fluorocarbons”* (Algrim, col. 5, lines 37-38, emphasis added) to prevent adhesion. Thus, according to Algrim, the silicone or fluorocarbons are the ingredient that prevents adhesion when applied to the

release material. Algrim teaches that it is the silicone or fluorocarbons that impart the non-adhesive characteristic when applied to the strip of (paper or polyester) release material. Algrim further states that after transport, when the shingles are applied to a roof, “[t]he release paper may be removed or left on during installation. . . .” (Algrim, col. 5, lines 29-32). Thus, the silicone or fluorocarbon-impregnated release material is useful for transport but may be removed prior to application of the shingle to a rooftop.

Fasold discusses coating the entire surface of the impregnated web with a weather resistant bituminous coating material. Fasold does not teach the coating of only a pressure point portion of the bottom surface. In fact, one of ordinary skill in the art viewing Fasold would conclude that the talc must be distributed along the entire surface of the shingle in order to optimize non-sticking, and thus, distributing it only along a pressure point would be inadequate for this purpose (and at the very least vastly inferior to the abilities of silicone or fluorocarbons).

One of ordinary skill in the art would not look at the talc taught in Fasold, which is to be applied to the entire surface of the roofing material, and combine it with Algrim to apply it to only the strip of release material. As stated previously, Algrim teaches a release material that is either paper or polyester, and that is treated (Algrim discusses several commercial suppliers of spray silicone and silicone-treated paper, at col. 5, lines 42-46). Algrim and Fasold together provide no teaching or suggestion that the talc of Fasold would adequately prevent sticking when applied directly to the shingle, and to only the “pressure point” portion of the shingle taught by Algrim. Thus, combining

Algrim with Fasold would, at best, teach one of ordinary skill in the art to apply talc to the entire surface, resulting in an increased cost of materials and labor.

Thus, combining Algrim with Fasold in the manner asserted by the Examiner would not result in the presently claimed invention, and moreover would render the shingle of Algrim unsatisfactory for its intended purpose. See MPEP § 2143.01; In re Gordon, 733 F.2d 900, 221 USPQ 1125 (Fed. Cir. 1984) (if proposed modification would render the prior art invention being modified unsatisfactory for its intended purpose, then there is no suggestion or motivation to make the proposed modification). Algrim teaches the treatment of the release material with silicone or a fluorocarbon for ease of transportation and storage; once the shingle is applied to a roof, the treated release material is optional. In contrast, the present claims are directed to application of a phyllosilicate directly to the surface of the shingle in a non-reversible manner. Once the phyllosilicate is applied to the shingle, its properties remain in the shingle through application to a rooftop. In order to arrive at the present invention, the combination of Algrim and Fasold would have to suggest getting rid of the release material entirely and applying a phyllosilicate only to that portion of the shingle itself. This would contravene the purpose of Algrim, which is to provide a shingle with a strip of release material that facilitates transport and storage without sticking, but which is of no value once the shingle is actually put onto a rooftop.

Even if the two references combined were to suggest the application of phyllosilicate to the pressure point (which Appellants argue is not the case), there would be no expectation of success in such a combination, because, *inter alia*, Algrim teaches

that other materials (no mention is made of phyllosilicates) are required when applied only to the strip of release material, and thus a skilled artisan would expect that application of talc to only the strip of release material would be inadequate. Even if the silicone or fluorocarbon taught by Algrim were completely replaced with the talc taught by Fasold, the resultant combination still would not yield the present invention, because the presently claimed invention requires adherence of phyllosilicate directly to the shingle and only to the pressure point of the shingle. Algrim and Fasold provide no teaching that this would be enough to prevent adhesion of the shingles during storage.

Appellants have discovered herein that application of the release coating only to the pressure point as shown in Fig. 2, not only results in a much greater ease of separation of the shingles from storage, but also results in significant savings of labor and material because the release coating need not be applied to the entire shingle to obtain this desirable result. Thus, such an application, as recited in the presently pending claims, is unexpectedly superior over the teachings of Algrim and Fasold.

Regarding the rejection of dependent claims 2, 3, 5-8 and 10, these claims are all dependent from, and incorporate the limitations of, independent claim 1. As set out above, the combination of the references does not disclose all the elements of independent claim 1. Accordingly, since the remaining dependent claims recite additional unique elements and/or limitations, these claims are also patentable over the cited references.

Thus, Appellants respectfully submit that claims 1-3, 5-8 and 10 are allowable under 35 U.S.C. § 103(a).

(8) CLAIMS APPENDIX

The text of the claims involved in the present appeal is as follows:

1. A roofing shingle comprising a top and bottom surface the bottom surface provided with a release coating of a continuous film of particles having good to perfect basal cleavage, wherein said release coating is disposed only on a pressure point portion of said bottom surface, wherein said particles are in the class phyllosilicates.
2. A shingle in accordance with claim 1 wherein said pressure point portion is immediately above and below a top edge of said bottom surface where the top edge overlaps a bottom portion of a headlap portion of said top.
3. A shingle in accordance with claim 1 wherein said roofing shingle is a shingle selected from the group consisting of a laminated shingle and a strip shingle.
5. A shingle in accordance with claim 1 wherein said particles are selected from the group consisting of Allophane, Apophyllite, Bannisterite, Carletonite, Cavansite, Chrysocolla, Baileychlore, Chamosite, Chlorite, Clinochlore, Cookeite, Nimite, Pennantite, Penninite, Sudioite, Glauconite, Illite, Kaolinite, Montmorillonite, Palygorskite, Pyrophyllite, Sauconite, Talc, Vermiculite, Delhayelite, Elpidite, Fedorite, Franklinfumaceite, Franklinphilite, Gonyerite, Gyolite and Leucosphenite; Biotite, Lepidolite, Muscovite, Paragonite, Phlogopite, Zinnwaldite, Minehillite, Nordite, Pentagonite, Petalite, Prehnite, Rhodesite, Sanfomite, Antigorate, Clinochrysotile, Lizardite, Orthochrysotile, Serpentine, Wickenburgite, Zeophyllite.



6. A stack of roofing shingles comprising a plurality of said roofing shingles of claim 1 wherein the top surface of each roofing shingle faces the bottom surface of its adjoining roofing shingle.
7. A stack of roofing shingles comprising a plurality of said roofing shingles of claim 2 wherein the top surface of each roofing shingle faces the bottom surface of its adjoining roofing shingle.
8. A stack of roofing shingles comprising a plurality of said roofing shingles of claim 3 wherein the top surface of each roofing shingle faces the bottom surface of its adjoining roofing shingle.
10. A stack of roofing shingles comprising a plurality of said roofing shingles of claim 5 wherein the top surface of each roofing shingle faces the bottom surface of its adjoining roofing shingle.

(9) EVIDENCE APPENDIX

- Exhibit A                    U.S. Patent No. 4,738,884 to Algrim et al., referred to on pages 2-4 of the Final Office Action and pages 2 and 3 of the Advisory Action
- Exhibit B                    U.S. Patent No. 2,326,724 to Fasold et al., referred to on pages 2-4 of the Final Office Action and pages 2 and 3 of the Advisory Action
- Exhibit C                    Wikipedia Entry for “Basal Cleavage” – obtained October 13, 2008, referred to on pages 2 and 3 of the Final Office Action and page 1 of the Advisory Action.

(10) RELATED PROCEEDINGS APPENDIX

None

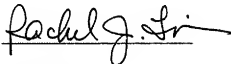
(11) CONCLUSION

For all the above reasons, Appellants submit that the appealed claims are patentable and respectfully request reconsideration and reversal of the rejection of the Final Office Action in view of the above arguments.

If there is any fee due not already accounted for, please charge such fee to Deposit Account No. 03-1250.

Respectfully submitted,

By:

A handwritten signature in black ink, appearing to read "Rachel J. Lin", written over a horizontal line.

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Exhibits

**Exhibit A – U.S. Patent No. 4,738,884 to Algrim et al.**

# United States Patent [19]

Algrim et al.

[11] Patent Number: 4,738,884

[45] Date of Patent: Apr. 19, 1988

[54] ASPHALT ADHESIVES SUPERIMPOSED  
ON ASPHALT-BASED ROOFING SHEET

[75] Inventors: Donald J. Algrim, Reynoldsburg;  
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Lamb, Granville; Stephen J. Jones,  
Newark, all of Ohio

[73] Assignee: Owens-Corning Fiberglas  
Corporation, Toledo, Ohio

[21] Appl. No.: 49,373

[22] Filed: May 14, 1987

## Related U.S. Application Data

[63] Continuation of Ser. No. 835,581, Mar. 3, 1986, abandoned.

[51] Int. Cl.<sup>4</sup> ..... B32B 3/06; B32B 11/04;  
B32B 27/04

[52] U.S. Cl. .... 428/57; 428/198;  
428/489; 524/68

[58] Field of Search ..... 524/68; 428/57, 198,  
428/489

## References Cited

### U.S. PATENT DOCUMENTS

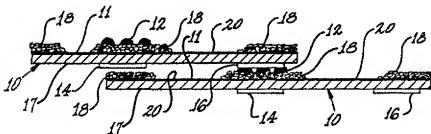
3,138,897	6/1964	McCordie	427/186
4,055,453	10/1977	Tajima et al.	428/291
4,196,115	4/1980	Breanon	524/68
4,217,259	8/1980	Breanon	524/68

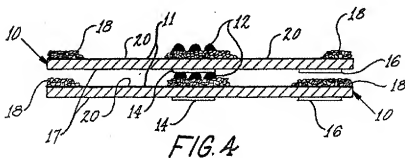
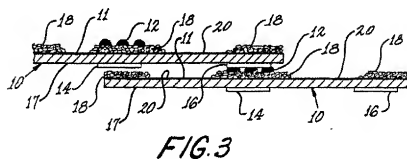
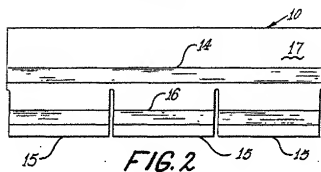
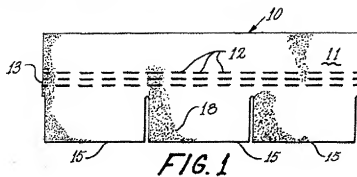
Primary Examiner—Allan M. Lieberman  
Attorney, Agent, or Firm—Patrick P. Pacella; Ted C.  
Gillespie

## ABSTRACT

An adhesive is provided for adhering roofing shingles wherein the adhesive is a blend of asphalt, an elastomer, a tackifying resin and a petroleum oil.

5 Claims, 3 Drawing Sheets





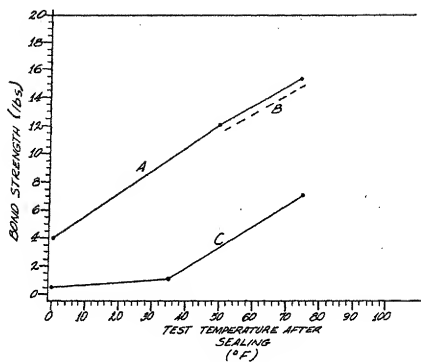


FIG. 5



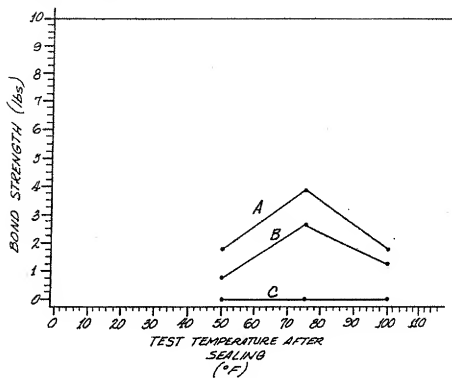


FIG. 6

# ASPHALT ADHESIVES SUPERIMPOSED ON ASPHALT-BASED ROOFING SHEET

This is a continuation of application Ser. No. 835,581 filed Mar. 3, 1986, abandoned.

## TECHNICAL FIELD

The present invention is related to asphaltic compositions, and more particularly to an asphalt adhesive for retaining shingles. The adhesive is a blend of asphalt, an elastomer, a tackifying resin and a petroleum oil. The present invention also relates to a roofing sheet or shingle employing this elastomer-modified asphalt adhesive to retain the tabs of shingles against windlift.

## BACKGROUND OF THE INVENTION

The use of adhesives, including asphaltic compounds, to provide a bond between roofing shingles when applied to a roof is known. During a typical shingle manufacturing process, a pattern of adhesive is applied to the headlap portion of the shingles so that the tab portion of the subsequently laid course of shingles on the roof will adhere to the headlap portion of the lower course, to help prevent wind uplift of the shingles. To seal properly, most adhesives or sealants require relatively high roof temperatures. U.S. Pat. No. 4,559,267 discloses an adhesive, of a compounded bitumen containing 3-20% rubber and/or thermoplastic resins, which requires an activation temperature of at least 90° F. Many other adhesives require roof temperatures of about 135° F. or higher. In relatively colder climates these roof temperatures may never be reached or in certain climates, these temperatures may not be reached until seasons subsequent to installation, which may be months later. Consequently, under conditions where relatively low temperatures do not permit proper sealing of the adhesive, the shingles may be susceptible to blow-off in relatively higher winds. Another problem with conventional sealants is that colder temperatures tend to cause the sealant on properly sealed shingles to become brittle and crack, resulting in bond failures and blow-offs.

U.S. Pat. No. 3,138,897 to McCorkle addresses the blow-off problem by using an adhesive strip on the shingle composed of distinct bands of two different adhesives one is pressure sensitive while the other is temperature sensitive. As with conventional adhesives, the temperature sensitive adhesive of McCorkle seals at relatively higher temperatures and since it doesn't even begin to get tacky until about 70° F., a second adhesive must be used to permit sticking at lower temperatures, which is the pressure sensitive adhesive. The pressure sensitive adhesive is effective only at lower temperatures since it loses its tackiness beyond temperatures of about 100° F.

An asphalt-based adhesive has now been discovered which is both pressure and temperature sensitive and effectively works to greatly reduce the vulnerability of a shingle to the cold and wind. The adhesive of the instant invention remains tacky at roof temperatures as low as 50° F. to provide a good initial bond upon shingle installation at these temperatures. While the adhesive seals the shingles at temperatures required by most sealants, i.e., 135° F. or higher, this adhesive also effectively seals the shingles at roof temperatures as low as 25° F. This means that air temperature may be as low as 25° F. Additionally, the adhesive retains appreciable strength and flexibility at lower temperatures which

means that the adhesive does not get brittle and crack and will not break an already formed seal.

A further advantage of having to apply only a single adhesive to the shingle is provided by the adhesive of the instant invention. The cost benefits of applying one sealant as opposed to two or more different sealants will become readily apparent to those skilled in the art, particularly when viewed from the standpoint of shingle manufacturing.

## STATEMENT OF THE INVENTION

According to this invention, there is provided an adhesive composition, for retaining the tabs of shingles against windlift at temperatures of about 50° F. and greater, comprising a blend of asphalt, an elastomer, containing about 80% triblock styrene-butadiene-styrene copolymer and about 20% diblock styrene-butadiene copolymer, a tackifying resin, and a petroleum oil.

According to this invention, there is also provided an asphalt roofing sheet having applied on at least one surface the above-described adhesive material, a contact surface and a release material.

In the broadest sense of the invention, it encompasses any asphalt-based roofing sheet employing the above-described adhesive, where the roofing sheet is of the type designed to be laid down in courses or layers, with at least a portion of successive sheets overlapping.

## BRIEF DESCRIPTION OF DRAWINGS

FIG. 1 is a plan view of the top side of a shingle with tab sealant adhesive;

FIG. 2 is a plan view of the bottom side of a shingle with a release surface and a contact surface;

FIG. 3 is a cross-sectional view of two shingles representing their relative positions upon installation.

FIG. 4 is a cross-sectional view of two shingles representing their relative positions in a package, before installation.

FIG. 5 is a graph of measured values for bond strengths of adhesives.

FIG. 6 is a graph of measured values for bond strengths of adhesives.

## DESCRIPTION OF THE INVENTION

The adhesive of the instant invention maintains sufficient tack at lower temperatures to provide a quick and good initial bond during installation and will seal shingles at roof temperatures as low as 50° F. when the air temperature may be as low as 25° F. Although the adhesive effectively seals at higher roof temperatures, it is especially useful for winter applications in colder northern climates and provides good resistance to blow-off.

The present adhesive uses an asphalt characterized by a kinematic viscosity in the range of from about 500 poise  $\pm$  100 to about 250  $\pm$  50 poise at 140° F. (60° C.) and a minimum viscosity of from about 110 cs (centistokes) to about 80 centistokes at 275° F. (135° C.). The asphalt can also be characterized by a penetration (ASTM D5 73) of from about 120 to about 300 dmm (deci millimeters) at 77° F. (25° C.). The asphalt of the instant invention exhibit a ring and ball softening point from about 90° F. to about 130° F.

Particularly good results were obtained with paving grade asphalt having a kinematic viscosity of about 500 poise  $\pm$  100 at 140° F. (60° C.), a minimum viscosity of about 110 cs at 275° F. (135° C.), a penetration of 120-175 dmm at 77° F., and a softening point from about 110° F. to about 120° F. These types of asphalt are

known as viscosity-graded asphalt or AC-5 paving grade asphalt which is commercially available from Amoco Chemical Corporation (Chicago, Ill., U.S.A.).

Also useful is an AC-2.5 grade asphalt, also commercially available from Amoco, which has been mixed with oil to achieve a blend of about 90% AC-2.5 asphalt and 10% oil. A suitable oil is one characterized as a soft flux oil having a kinematic viscosity at 210° F. of about 60-90 cs which is commercially available from Marathon Oil Company (Findlay, Ohio, U.S.A.) and known as 432 oil. The asphalt blend is characterized by a softening point of about 100°-110° F., a penetration of from about 250-300 dmm at 77° F. and a viscosity of about 250±50 poise at 140° F.

The elastomers of the present invention are thermoplastic and selected for their ability to impart strength to the adhesive at colder temperatures. As with conventional thermoplastic organic polymers, these elastomers can be processed, i.e., melted and extruded, and can be repeatedly heated and cooled with no substantial loss in their properties, especially their elastomeric properties. Therefore, the elastomers employed herein substantially retain their properties when subjected to heating and cooling cycles. Particularly desirable is the retention of strength upon cooling the elastomer which gives strength and flexibility to the sealant at colder temperatures.

The elastomers employed in the present invention are block copolymers, usually triblock (A-B-A) and may be linear or radial in structure. Either block, A or B, may comprise more than one monomer. Preferred are those triblock copolymers having styrene or polystyrene as the "A" block or end block units. Suitable elastomers include thermoplastic rubbers of styrene-butadiene-styrene (S-B-S), styrene-isoprene-styrene (S-I-S) and styrene-ethylene-butylene-styrene (S-E-B-S) block copolymers. Preferred is a styrene-butadiene-styrene block copolymer, and especially one containing about 80% styrene-butadiene-styrene triblock copolymer and about 20% styrene-butadiene diblock copolymer. Suitable elastomers are commercially available from the Shell Chemical Company (Houston, Tex., U.S.A.) as Kraton® thermoplastic rubbers, Kraton D and Kraton Q grades. Most preferred is Shell's Kraton D-1101 (S-B-S) rubber product which is a linear triblock copolymer containing about 80% triblock styrene-butadiene-styrene copolymer and about 20% diblock containing about 31% styrene and 69% butadiene, and which has a nominal molecular weight of about 100,000.

The tackifying resin can be any resinous material recognized in the art as enhancing the tack of the adhesive composition. Desirably, tackifiers will also impart cohesive strength or body to the adhesive so as to make it firm and not too soft. Suitable tackifying resins include rosin, rosin derivatives, polyterpene resins, thermoplastic phenolic resins, hydrogenated rosin esters of pentaerythritol, cumaronendene and the like. Particularly good results were obtained using a modified hydrocarbon resin commercially available from the Neville Chemical Company (Pittsburgh, Pa., U.S.A.) known as Nevprene® (9500 Tackifying Resin. Other suitable tackifiers commercially available include terpene resins called Wingtack®, from the Goodyear Tire & Rubber Co. (Akron, Ohio, U.S.A.) and Piccolite® from Hercules Chemical Company (Wilmington, Del., U.S.A.). It will be appreciated by those skilled in the art that the particular tackifier selected may vary

with the specific asphalt used in order to achieve the desired properties of the final adhesive.

The petroleum oil used herein is the resinous by-product of a lubricating oil tower used in the crude oil refining process. Generally, in the oil refining process, a mixture of volatile hydrocarbons is separated from an asphaltic residue. One subsequent treatment of this residue is to further process it in a lubricating oil tower to yield a light fraction high in heterocyclic hydrocarbons and another residue. This residue is a petroleum oil generally characterized as being relatively soft and high in resins. When used in the instant invention, this petroleum oil is believed to aid in holding the other components together and to impart a tacky characteristic to the sealant. Another desirable characteristic of this resin-containing petroleum oil is its thermal stability. Without being limited as to theory, it is believed that this petroleum oil compatibilizes the system to help prevent phase separation. This petroleum oil is also believed to improve the tackiness of the adhesive at lower temperatures. This material is commercially available as Hub P-Resin from Borcke Associates, Inc. (Great Neck, N.Y., U.S.A.). Hub-P resin is characterized by a viscosity at 210° F. of 2300/2800, a pour point in °F. of +85, an acid number of about 0.15, and contains about 0.10% hard asphalt, 0.15% sulphur and 12.0% carbon residue.

Conventional mixing or blending techniques may be used to make the sealant. Generally, throughout the mix, the temperature is desirably maintained from about 260° F. (126.6° C.) to about 360° F. (182.2° C.). Typically, the adhesive is cooled for packing and then melted for application to a shingle. It may be desirable to circulate and maintain the adhesive at an elevated temperature during processing and application to the shingles to aid in the prevention of phase separation.

Satisfactory results have been obtained when the ingredients of the sealant are present in an amount, in approximate weight percent, of about 25% to about 80% asphalt, about 3% to about 18% elastomer, about 5% to about 25% tackifying resin, and about 10% to about 50% petroleum oil. Preferably, the sealant contains from about 35% to about 60% asphalt, from about 5% to about 12% elastomer, from about 8% to about 20% tackifying resin and from about 15% to about 35% petroleum oil. The most preferred composition is one consisting essentially of, in approximate weight percent, 42% to 48% paving grade asphalt, 10% to 11% elastomer, 17% to 19% tackifying resin and 22% to 28% petroleum oil.

The present invention also provides a roofing shingle employing the above-described adhesive.

In the broadest sense of the invention it encompasses any asphalt-based roofing sheet employing the above-described adhesive, where the roofing sheet is of the type designed to be laid down in courses or layers, with at least a portion of successive sheets overlapping. The invention in the form of an asphalt roofing membrane solves sealing problems by providing good seal at cold temperatures for the overlapping portions of a newly laid down asphalt roofing membrane.

With reference to the drawings, the preferred embodiments, FIG. 1 shows the top surface 11 of a shingle 10 having the tab sealant adhesive 12 applied in the headlap portion 13 of the shingle. The shingle 10 can be any conventional shingle known in the art. Particularly suitable shingles are those made of asphalt reinforced by glass fibers, as exemplified by U.S. Pat. No. 3,332,830,

herein incorporated by reference. The adhesive is preferably applied to the headlap portion 13 of the shingle and holds down the overlying tabs 15 of a shingle in the next upper row when installed on a roof. Although FIG. 1 shows the adhesive 12 applied as three discontinuous strips, the adhesive can be applied in any form or configuration which provides an adequate surface area for adhering an overlying shingle. For example, the adhesive may be applied as one continuous strip, or any combination of a number of continuous and/or discontinuous strips of varying dimensions. The sealant may also be placed anywhere on the shingle which would be effective in adhering overlapping shingles, including the bottom side of the shingle.

As shown in FIG. 3, the top surfaces 11 of the shingles are typically covered with granules 18 of crushed rock, and the adhesive 12 is applied over the granules 18.

FIG. 2 shows the bottom surface 17 of a shingle 10 having a strip of release material 14 a strip of contact surface 16 on the shingle tab 15. Although this location represents the preferred embodiment, the release material 14 and the contact surface 16 may be located on the top surface 11 of a shingle. When the strip of release material 14 is located on the bottom surface 17 of the shingle in a position which corresponds to the position of the strip of tab sealant adhesive 12 on the top surface 11, as shown in FIG. 4, the shingles are prevented from sticking together during packing where they are usually stacked upon each other. The release paper may be removed or left on during installation without any adverse effect on the performance of the shingle.

The release material can be of any material which does not adhere to the sealant so as to prevent the shingles from sticking to each other, particularly before installation. Suitable release materials include paper or polyesters which have to be treated with a non-adhering substance such as silicone or fluorocarbons. Alternatively, the release material may be a liquid or emulsion of silicone- or fluorocarbon-based substances which are applied directly to the shingle by any method, including spraying. Silicone-treated paper is commercially available from James River Corporation (Parchment, Mich., U.S.A.) and a silicone-based emulsion for spray applications is commercially available from Paper-Chem Labs (Rockhill, N.C., U.S.A.).

As shown in FIG. 3, the contact surface 16 works together with the adhesive 12 to form an extra-tight bond between overlapping shingles after installation. The location of the contact surface 16 on the bottom surface 17 one shingle 10 corresponds to the position of the tab sealant 12 on the top surface 11 of the underlying shingle 10 to form a tight bond between shingles upon installation.

The contact surface 16 may be covered with any material to which the adhesive will adhere, especially in colder temperatures. Suitable materials include polyester, polypropylene, polyethylene, polybutylene, a copolymer of polyethylene and vinyl acetate and may be applied in any form, including strips, films, liquids or emulsions. Preferred is a polyester film commercially available as Mylar® from E. I. DuPont de Nemours & Co. (Wilmington, Del., U.S.A.).

#### SPECIFIC EMBODIMENTS

##### EXAMPLE 1

The following experiment was conducted to test the bond strength of adhesives after shingles bearing the

adhesives were sealed at about 135° F. The bond strength test was conducted by sealing, at 135° F. for 16 hours, two overlapping pieces of roofing shingles bearing various adhesives. Upon cooling, the bond strengths of the adhesives were measured at various temperatures. To measure the bond strengths of the adhesives, an Instron tensile pulling machine, or equivalent apparatus, was used. The machine permits the bottom and top shingle sections to be clamped into place and then pulled while a load cell attached to the upper clamp measures the amount of force required to pull the shingles apart, which is recorded in units of pounds.

Three asphaltic adhesives were tested for bond strength using this method and are identified in Table I. Adhesives A and B represented formulas of the instant invention while adhesive C was a standard commercially available asphaltic adhesive known as Seal Rite™, commercially available from Owens-Corning Fiberglass Corporation (Toledo, Ohio, U.S.A.).

TABLE I

Adhesive	Content
A	asphalt, s.p. 110° F.-120° F. elastomer tackifying resin petroleum oil
B	asphalt, s.p. 100° F.-110° F. elastomer tackifying resin petroleum oil
C	asphalt approx. 60% propane washed approx. 40% roofing grade

The results are summarized in FIG. 5, which is a graph depicting the measured bond strengths of adhesives A, B and C represented by lines A, B and C, respectively. Each data point on the graph represents a value which is the average of values obtained from several tests under similar conditions. The bond strength values obtained for adhesive B at 50° F. and 75° F. were the same values obtained for adhesive A at these temperatures. Line B is depicted as a separate dashed line for purposes of clarity in presenting the data.

As can be seen from the test results, the adhesives of the instant invention retained substantially greater bond strength as compared to the standard adhesive at 50° F. when the temperature of the shingles was reduced after sealing at 135° F.

##### EXAMPLE 2

The above adhesives were also tested according to the Underwriter's Laboratory wind test UL 997 for shingles. To conduct the test, shingles bearing the adhesive were stapled to a plywood deck measuring about 54 in. by 4 ft. The shingles were then sealed in an oven at a temperature of about 135-140° F. for about 16 hours. After the deck cooled to room temperature, it was placed at a 4 in 12 slope and a 60 mph wind was blown on the deck. It was found that after 2 hours, no tabs lifted on shingles bearing adhesives A and C, while 3 tabs lifted after 45 minutes on shingles bearing adhesive B. Consequently, the inventive adhesive containing the harder asphalt (Adhesive A) provided better resistance than the inventive adhesive with the softer asphalt (Adhesive B) against the winds encountered in the Underwriter's Laboratory wind test.

## EXAMPLE 3

An experiment was conducted to test the bond strength of adhesives at the same temperature at which shingles bearing the adhesive were sealed.

To test the adhesive, the shingles were placed together and allowed to adhere at testing temperature for a period of about 16 to 24 hours. At the same temperature, the bond strength of the adhesive was tested using the same apparatus and testing technique described in Example 1. When the testing temperature was below room temperature, i.e., 50° F., the shingles were cooled for 1 hour at 50° F. before sealing them.

The same three adhesives, A and B of the invention and C, a standard adhesive, as in Example 1, were 15 tested.

The results are summarized in FIG. 6 which is a graph depicting the measured bond strengths of adhesives A, B and C, represented by lines A, B and C respectively, according to the procedure described above. 20 Each data point on the graph represents a value which is the average of values obtained from several tests under similar conditions.

As can be seen in FIG. 6, the inventive adhesives, A and B, provided especially good initial cold-temperature bonding strength at 50° F. as compared to the standard adhesive, C, which demonstrated no bond strength at 50° F., 75° F. and 100° F.

Although the invention has been described in terms of specific embodiments of a manner the invention may 30 be practiced, this is by way of illustration only and the invention is not necessarily limited thereto since alternative embodiments and operating techniques will become apparent to those skilled in the art. Accordingly, modifications are contemplated which can be made 35 without departing from the spirit of the described invention.

We claim:

1. An asphalt-based roofing sheet having superposed thereon an adhesive composition, for retaining the tabs 40 of shingles against windlift at temperatures of about 50° F. and greater, a contact surface, and a release material, and wherein said adhesive is one comprising a blend of asphalt, an elastomer containing about 80% triblock styrene-butadiene-styrene copolymer and about 20% 45 diblock styrene-butadiene copolymer, a tackifying resin and a petroleum oil;

wherein the blend contains about 25-80% asphalt, 3-18% elastomer, 5-25% tackifying resin and 10-50% petroleum oil;

wherein the asphalt is characterized by a kinematic viscosity in the range of from about 500 poise  $\pm 100$  to about 250  $\pm 50$  poise at 140° F. (60° C.), a minimum viscosity of from about 110 cs (centistokes) to about 80 centistokes at 275° F. (135° C.), 55 a penetration (ASTM D5 73) of from about 120 to about 300 dmm (decimillimeters) at 77° F. (25° C.), and a ring and ball softening point from about 90° F. to about 130° F.; and

wherein the petroleum oil is a resinous by-product of a lubricating oil tower used in the crude oil refining process.

2. A roofing sheet having tabs, a headlap portion, a top surface and a bottom surface, where said top surface has applied thereon an adhesive in the headlap portion 65 of said shingle, and said bottom surface has a release

material in said headlap portion of the shingle and a contact surface on the tabs, wherein said adhesive seals said sheets at 50° F. and greater and said adhesive is a blend comprising asphalt, an elastomer containing 5 about 80% triblock styrene-butadiene-styrene copolymer and about 20% diblock styrene-butadiene copolymer, a tackifying resin and a petroleum oil;

wherein the asphalt is characterized by a kinematic viscosity in the range of from about 500 poise  $\pm 100$  to about 250  $\pm 50$  poise at 140° F. (60° C.), a minimum viscosity of from about 110 cs (centistokes) to about 80 centistokes at 275° F. (135° C.), a penetration (ASTM D5 73) of from about 120 to about 300 dmm (decimillimeters) at 77° F. (25° C.), and a ring and ball softening point from about 90° F. to about 130° F.; and

wherein the petroleum oil is a resinous by-product of a lubricating oil tower used in the crude oil refining process.

3. A pair of superimposed shingles partially overlapped to form an overlapping area in which one shingle has applied on its surface an adhesive composition and the other shingle bears a contact surface for adhering said adhesive, and wherein said adhesive is a composition for retaining the tabs of said shingles against windlift at temperatures of about 50° F. and greater and said adhesive is a blend comprising asphalt, an elastomer containing about 80% triblock styrene-butadiene-styrene copolymer and about 20% diblock styrene-butadiene copolymer, a tackifying resin and a petroleum 10 oil;

wherein the asphalt is characterized by a kinematic viscosity in the range of from about 500 poise  $\pm 100$  to about 250  $\pm 50$  poise at 140° F. (60° C.), a minimum viscosity of from about 100 cs (centistokes) to about 80 centistokes at 275° F. (135° C.), a penetration (ASTM D5 73) of from about 120 to about 300 dmm (decimillimeters) at 77° F. (25° C.), and a ring and ball softening point from about 90° F. to about 130° F.; and

wherein the petroleum oil is a resinous by-product of a lubricating oil tower used in the crude oil refining process.

4. An asphalt-based roofing sheet having superposed thereon an adhesive composition for adhering the roofing sheet to an adjacent roofing sheet, a contact surface, and a release material, and wherein said adhesive is one comprising a blend of asphalt, an elastomer containing about 80% triblock styrene-butadiene-styrene copolymer and about 20% diblock styrene-butadiene copolymer, a tackifying resin and a petroleum oil;

wherein the blend contains about 25-80% asphalt, 3-18% elastomer, 5-25% tackifying resin and 10-50% petroleum oil;

wherein the asphalt is characterized by a kinematic viscosity in the range of from about 500 poise  $\pm 100$  to about 250  $\pm 50$  poise at 140° F. (60° C.), a minimum viscosity of from about 110 cs (centistokes) to about 80 centistokes at 275° F. (135° C.), a penetration (ASTM D5 73) of from about 120 to about 300 dmm (decimillimeters) at 77° F. (25° C.), and a ring and ball softening point from about 90° F. to about 130° F.

5. The roofing sheet of claim 4 comprising an asphalt roofing membrane. . . . .

**Exhibit B – U.S. Patent No. 2,326,724 to Fasold et al.**

Aug. 10, 1943.

G. A. FASOLD ET AL

2,326,724

ROOFING

Filed June 20, 1941

3 Sheets-Sheet 1

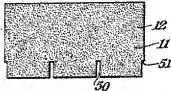
*Fig. 1.*



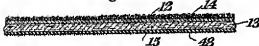
*Fig. 2.*



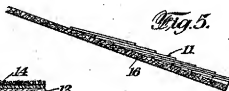
*Fig. 3.*



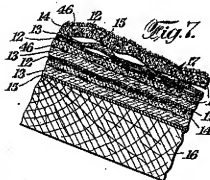
*Fig. 4.*



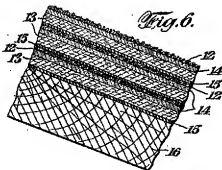
*Fig. 5.*



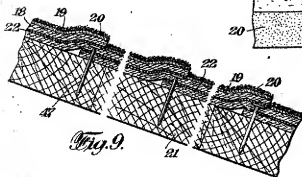
*Fig. 7.*



*Fig. 6.*



*Fig. 9.*



*Fig. 8.*



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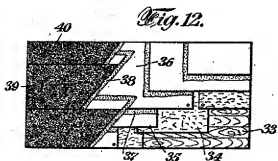
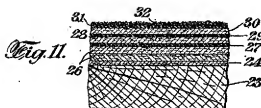
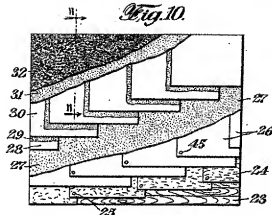
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2,326,724

ROOFING

Filed June 20, 1941

3 Sheets-Sheet 2



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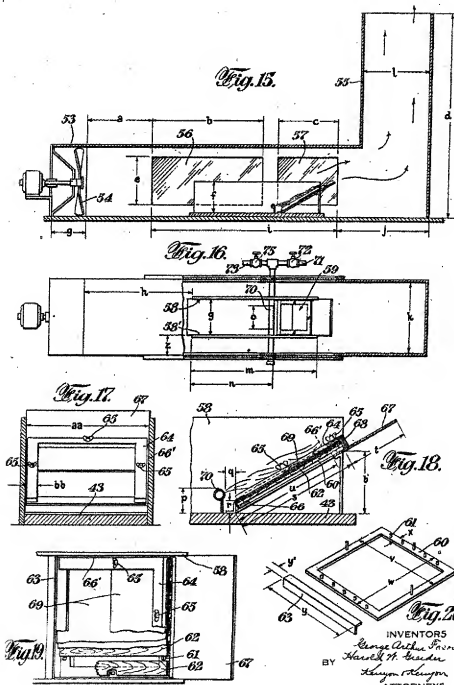
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2,326,724

ROOFING

Filed June 20, 1941

3 Sheets-Sheet 3



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## UNITED STATES PATENT OFFICE

2,326,724

## ROOFING

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Application June 20, 1941, Serial No. 399,824

15 Claims. (Cl. 154—51)

This invention relates to roofing and the manufacture thereof and is a continuation in part of our application Serial No. 347,154 filed July 24, 1940. This invention relates particularly to roofing embodying bituminous material as a waterproofing. While reference is made to "roofing," this term is used generally as referring to water- and weather-resistant coverings such as shingles (individual or strip shingles), roll roofing, cap sheets, sidings, roof deck coverings made from such preformed materials, built up roofings and the like.

Heretofore bituminous prepared roofing has been very extensively manufactured using as a base a fibrous web such as a sheet of roofing felt, impregnating the fibrous web with a bituminous material, and coating one or both surfaces of the impregnated web with a weather-resistant bituminous coating material. The bituminous coating material usually contains a mineral filler such as slate flour or powdered limestone. Usually there is applied to the bituminous coating on the surface intended to be exposed to the weather a suitable granular material such as slate granules or finely divided mineral surfacing material such as talc or mica. Finely divided materials such as mica flakes, talc, silica dust or the like may be made adherent to the non-weather exposed surface of the roofing to prevent sticking of the adjacent layers of the roofing material in the package.

According to the present invention roofings, for example roofings of the character referred to, can be improved to a very pronounced degree as will appear more in detail hereinafter. An important object of the present invention is to make roofing comprising bituminous waterproofing material so resistant to the common defect of blistering that it may be exposed to very severe conditions of summer sun temperatures and moisture without harmful blistering tendency.

According to this invention roofing can be made which not only is greatly improved as to blistering resistance but also is greatly improved in fire resistance, and the manufacture of roofings having both high blister resistance and high-fire resistance is also an important feature and object of this invention. While improvement in fire resistance is not a necessary incident to all embodiments of this invention, those embodiments which have improved fire resistance are regarded as preferred embodiments of the present invention. The importance of high fire resistance is self-evident. According to the present invention a roofing of the relatively inexpensive

bituminized felt type can be made which is comparable in fire resistance to more costly non-bituminous types of roofing such as slate and asbestos cement shingles. In our pending application Serial No. 370,636 filed December 18, 1940, for Roofing and material for roofing and the manufacture thereof, we have described roofings and roof coverings which are primarily characterized by their high fire resistance and which include embodiments that do not necessarily have the high blister resistance of the present invention. This application in certain respects likewise is a continuation in part of said application Serial No. 370,636.

It is another important aspect of this invention that the blister prevention and also fire resistance can be obtained without sacrifice of other desirable attributes of the roofing and that in preferred embodiments of this invention the roofing can be improved in respects other than blister resistance and fire resistance. Thus, roofings, having a bituminized fibrous web base and that embody this invention have been made which exhibit greatly improved strength in comparison with roofings of this type heretofore made as measured by such well known tests as the Mullen bursting test and the tensile strength test. Moreover, roofings embodying this invention, e. g., in the form of shingles, can be made which are considerably tougher and more resistant to impact shocks than ordinary roofings heretofore made and which likewise have greater physical stability under conditions of exposure to weather, are less subject to deteriorative changes, and have improved resistance to sliding. It is a further advantage of this invention that roofings may be made which have improved temperature susceptibility, that is, which exhibit less change in properties with temperature changes than do ordinary bituminized roofings heretofore made. According to preferred embodiments of this invention, roofings are afforded which, while somewhat stiffer than ordinary bituminous roofings heretofore made at normal atmospheric and at roof temperatures, are more pliable and flexible without rupture at lower temperatures such as 50° F. and even at 32° F. Moderate stiffness at ordinary atmospheric temperatures and at roof temperatures is a desirable quality, but excessive brittleness and lack of pliability either at normal atmospheric temperatures or at low temperatures such as occur when a roofing becomes hard and brittle upon prolonged ageing are objectionable.

According to this invention the blistering prob-

lem is remedied by providing a bituminous layer or coating for the roofing which is of such strong nature at summer sun roof surface temperature as to successfully resist the formation of blisters due to the vapor pressure of entrapped moisture at such temperatures and which at the same time has the other qualities and characteristics that are desirable in a bituminous roofing and even improves upon such other qualities and properties, e. g., in the respects mentioned above. This represents a radical departure from ordinary bituminous roofings which carry a coating of asphalt that especially under summer sun temperatures is highly plastic and is readily deformed and blistered by any moisture that becomes entrapped in void spaces of the roofing.

Heretofore blistering has for many years been a serious defect of bituminous roofings and numerous attempts have been made by technologists in the industry to remedy blistering. So far as we are aware the roofings that have been made heretofore which afford the highest resistance to blistering are roofings that have been manufactured utilizing the method and apparatus which we have disclosed in our United States Patents Nos. 2,105,531 and 2,159,587, the improved product of high resistance to blistering and high durability being covered by our Patent No. 2,159,586, and roofings embodying the inventions of these patents have been manufactured and sold in very large quantities. In these prior patents we have pointed out how the porosity of a bituminous roofing and particularly the porosity of a base sheet such as roofing felt, can be reduced to an exceedingly small percentage thereby minimizing the blistering tendency. While the present invention may be utilized in conjunction with the inventions covered by our aforesaid patents and in preferred practice is so used, this is not necessarily the case inasmuch as exceedingly high resistance to blistering can be afforded by different means, namely, by providing a coating which is so strong at summer sun roof surface temperatures that it acts as a blister barrier through which blisters cannot push from beneath and in which blisters cannot form and without utilizing the inventions of our prior patents.

In those embodiments of this invention which have high fire resistance, the fire resistance is due to the composition and characteristics of the bituminous coating composition or waterproofing layer. While the base sheet for the roofing might be made of non-combustible material this does not solve the problem if the bituminous waterproofing coating itself burns or tends to spread the flame. One of the attributes of the coating composition which render it fire resistant is that the coating while comprising an essentially thermoplastic bituminous base also comprises a finely divided heat resistant material having surface characteristics such that as incorporated in the bituminous coating it is flow resistant when the bitumen in the coating becomes softened by heating to elevated temperatures at or approaching flame temperatures and provides a skeletal mat which remains coherent and persists in place so that the finely divided heat resistant material does not tend excessively to flow down an inclined roof deck, e. g., a roof deck having a 30 degree inclination to the horizontal, having underlying material such as a combustible organic felt base exposed to the flame. Another attribute of the roofing is that the fire resistant coating when exposed to high temperatures approaching or at flame temperatures tends to re-

sult in a coherent mat-like mass which has high heat insulating properties and in preferred embodiments develops pores therein, augmenting the heat insulating effectiveness thereof so as to shield an underlying combustible roof deck from the heat of the flame. Another attribute of the fire resistant coating is that it is highly resistant to combustion so that any charring upon exposure to flame is gradual and discontinuous without substantial spreading as soon as exposure to flame is discontinued and preferably the coating when subjected to flame temperatures is substantially non-bleeding, that is, the bitumen does not tend to separate and flow from the skeletal mat of heat resistant material but chars and carbonizes while remaining in place commingled with the finely divided heat resistant material.

Other features of this invention relate to the disposition of the special coating material in strata in roof deck coverings and to the disposition, character and quantity of the finely divided mineral filler in the coating material and in the roofing structure.

In order to afford a better understanding of the practice of this invention it will be described in connection with a specific example of the practice thereof which is both blister resistant and fire resistant. The material that is used for the blister barrier and fire resistant surface coating or layer will first be described. A typical bituminous base for this material is obtained by air-blowing a residual asphalt flux from the refining of Mid-Continent petroleum to a softening point, ring-and-ball method, of about 220° to 240° F. The asphalt is heated to a heat-liquefied condition, and into the heated asphalt is incorporated about 40% of "asbestos dust." (The percentage being by weight based on the total weight of the mixed asphalt and finely divided asbestos.)

The finely divided asbestos in the form of asbestos dust is a by-product of the chrysotile asbestos milling industry and is characterized by being of very short fibre length and small particle size. A typical grade of asbestos dust is that produced by the Quebec Asbestos Corp., of East Broughton, P. Q., Canada. The asbestos dust referred to in the present example is a dust that about 85% by weight passed a 48 mesh standard testing sieve, about 65% passed a 100 mesh sieve, about 50% passed a 200 mesh sieve, and about 35% passed a 325 mesh sieve. This material has heretofore had no commercial use and has been regarded as worthless waste.

The bituminous mass containing the finely divided asbestos even at high mixing temperatures (about 400° to 500° F.) exhibits a certain physical stability and resistance to flow and exhibits semi-plastic characteristics as distinguished from being a highly fluid mass. Notwithstanding this semi-plastic state we have found that the mass can readily be applied to a suitable foundation by a coating operation. At temperatures of about 390° to 500° F. the high viscosity mass applies easily and bonds well to the base. At temperatures below about 390° F. the mass becomes somewhat difficult to work and does not spread as satisfactorily.

The base material that is used according to the present example of the practice of this invention is ordinary roofing felt made of vegetable and animal fibre, e. g., roofing felt weighing about 8 pounds per 100 square feet. The felt is impregnated with a suitable bituminous impregnating material, e. g., a conventional bituminous roofing saturant having a softening point of about 110°

F. to about 160° F. The roofing felt may be impregnated with the bituminous saturant and coated with the special coating composition using any conventional type of impregnating and coating equipment. The felt base sheet as thus composed is porous and water absorptive and when exposed to fire is consumed quite readily.

Preferably, and for the purpose of the present example, the method and apparatus disclosed in our Patents Nos. 2,105,531 and 2,159,587 are employed for the purpose of impregnating the felt base in order to obtain maximum blister resistance and durability as well as maximum fire resistance. In so doing the felt is first thoroughly impregnated with bituminous saturant as by passing it a plurality of times through a bath of the saturant that is maintained at a temperature of about 350° to 400° F. After the felt is thoroughly impregnated with the saturant it is immersed in a bath of special bituminous coating composition above described, maintained at a temperature of about 450° to 500° F. and, as disclosed in our aforesaid prior patents, is squeezed between squeeze rolls to expel residual air and vapors and is caused to become sealed with the coating composition while the felt is devoloided and before the felt can reabsorb air or vapor. After removing the impregnated felt that has been filled with the impregnating material, devoloided, and sealed in a devoloided condition, from the bath of special bituminous coating composition, an additional coating of the special coating composition is applied to the surface of the roofing that is to be exposed to the weather so as to provide a surface layer of the coating composition that is substantially uniformly distributed and weighs about 50 pounds per 100 square feet of the roofing. The fineness of subdivision of the asbestos dust in the coating composition enables the composition to be spread to uniform thickness and with uniformity of distribution of the asbestos dust in the coating. As the roofing cools conventional mineral surfacing material such as slate granules can be applied to the surface for weather exposure and the granules can be partially embedded in the coating composition. The granular surfacing material is preferably substantially non-porous and is preferably substantially non-combustible. On the opposite side of the sheet there may be applied a thin coating of bituminous coating material, e. g., having a softening point of about 220° F. P. so as to weigh about 5 pounds per 100 square feet and a finely divided dusting material such as talc or mica dust or similar material may be applied and partially embedded in the coating. Preferably for maximum fire resistance the back coating should be special coating composition embodying our invention, e. g., the same as applied to the top or weather side of the sheet. Because of the relatively high temperatures employed in applying the coating material it is desirable to provide somewhat more cooling means than are customarily employed for cooling the roofing so that the machine may be operated at normal speed and so that the finished roofing will emerge at a temperature which is not excessively high and at which cutting and packing in the regular way are permitted.

In the special coating as applied it is important that the asbestos dust be disposed uniformly and continuously both in amount and distribution over the base sheet. To this end the special coating material should be thoroughly mixed and should be applied in a thoroughly mixed condition to the base sheet. A suitable

agitator in the reservoir for the mixed coating composition can be used for this purpose. We have found that the mixing of the asbestos dust with the bitumen can be facilitated by preheating it to at least about 350° F. before it is mixed with the bitumen. Preferably the finely-divided asbestos is heated to a temperature above the temperature at which the heat liquified bitumen is maintained so that during mixing the bitumen is brought up to a desirably high mixing temperature due to the heat of the filler. For example, we have found it to be desirable to maintain bitumen in storage at about 400° F. and to preheat the finely-divided asbestos to about 500° F. When the bitumen is taken from the reservoir to the mixer it is mixed with the asbestos dust which raises the temperature of the mixture to about 425° F. which is suitable for the mixing step. The mixed material is then taken to the reservoir which supplies the coater and in which the material is agitated to maintain homogeneous distribution of the asbestos dust until immediately prior to the coating step. By following the preferred procedure the bitumen can be handled and mixed without local overheating and the stiffening effect of the finely-divided asbestos interferes to a minimum extent with the mixing operation. Moreover, this procedure facilitates the application of the coating composition in a very uniform layer on the felt and in a condition such that the finely-divided material will become distributed continuously and uniformly both in amount and distribution over the felt. The foregoing preferred procedure is likewise preferable when finely-divided mineral fillers other than asbestos dust are used in the coating composition.

The roofing as thus prepared weighs about 105 pounds per 100 square feet of sheet area. Of this weight about 25% is in the form of slate granules adherent to one side and finely-divided dusting material adherent to the other side and the balance of the weight is the felt, the bituminous impregnating material and the special coating material. Because of the employment of the method and apparatus of our Patents Nos. 2,105,531 and 2,159,587 the roofing as a whole contained less than about .5% of voids and the coating composition was sealed to the sides of the devoloided felt and was integrally bonded with the bituminous saturant in the felt and with the fibers adjacent the surface of the felt and filled the surface interstices of the felt to substantial depth without substantial porosity. Moreover, the asbestos dust was keyed into the surface interstices of the felt and anchored thereto as a mat and the amount was reduced of asphalt in the special coating composition that might bleed to the surface and tend to promote combustion.

The roofing as thus prepared exhibited many remarkable properties, a highly important property being its exceedingly great resistance to blistering. Blistering of an asphalt roofing is a defect which may appear very soon after application to the roof or it may develop gradually with increase in the number and size of the blisters over a period of two to four years. The blistering occurs as bubbles formed in or under the coating as a result of stretching or deformation or movement of the coating material resulting from the pressure exerted by entrapped air and moisture at summer sun roof surface temperature. In order to test bituminous roofings for blistering tendency various accelerated blistering tests have been devised by bituminous

roofing technologists which give a fairly accurate indication of blistering tendency. These methods have included (a) soaking the roofing specimen in water at ordinary temperature or at an elevated temperature such as 125° F. to 140° F. followed by heating in an oven at a still higher temperature such as 175° F. or 220° F.; (b) heating the specimen in an oven to expel air, immersing it in water, and heating it again in an oven; (c) exposure of the specimen to repeated accelerated weathering cycles of exposure to actinic light, heat, wetting and drying, intense water spray and freezing, as in a Weather-Ometer; and (d) soaking a plurality of test specimens in water for varying periods of time followed by heating at 220° F., the minimum soaking time required to induce blistering being recorded.

The roofing prepared as above described has been subjected to the foregoing tests, but we have found that these tests are not sufficiently severe to show the very great improvement in blister resistance of the new product as compared with asphalt roofings made according to prior art practice. We therefore have devised a blister resistance test that is still more severe, and which consists simply in immersing roofing specimens in boiling water and noting the time that the specimens may be immersed without blistering. The drastic nature of this test is apparent when it is considered that commercial asphalt prepared roofings produced by ten of the largest manufacturers thereof blistered badly after only 30 to 60 minutes' immersion in boiling water and several blistered badly after immersion of merely 15 minutes. By contrast, the roofing made as above described could be immersed in boiling water up to eight hours and even longer and remain virtually blister-free. This makes it apparent that according to the present invention roofing can be manufactured which exhibits a very remarkable improvement in blister resistance.

The boiling water test is indicative of the capacity of the surface coating or layer as a whole to resist without blistering the vapor pressure at boiling temperatures of any air and moisture that may be entrapped on or in a fibrous backing sheet and that tends to push out through the bituminous coating. The test is also indicative of the capacity of the surface coating or layer to resist absorption of water or moisture by the coating itself and penetration of water or moisture into the coating for, if the coating were to absorb substantial quantities of water or moisture, the absorbed water or moisture would form blister sources intermediate the thickness of the coating. We have found that to afford blister resistance to the highest degree, the blister barrier layer or coating should be of such high viscosity and strength as to resist without substantial deformation vapor pressure generated at summer sun roof surface temperatures of moisture entrapped underneath the layer or coating and the layer or coating should also be substantially non-water absorptive and sealed on the interior from access

of water. At summer sun roof surface temperatures, any moist air entrapped in the roofing develops a definite vapor pressure in excess of atmospheric pressure unless the pressure is relieved by escape of moisture vapor or by evaporation. Summer sun roof deck temperatures vary according to localities depending upon the climate, although in the mid-summer months roof surface temperatures in all parts of the United States are well above

normal atmospheric temperature if there is open exposure to the sun. The most severe conditions in the United States that tend to cause blistering occur in the central gulf coastal area where there is high humidity combined with high roof temperatures which frequently attain 160° F., although they seldom exceed 150° F. In a few instances, roof surface temperatures as high as 180° F. have been recorded.

At temperatures such as 170° F., any moisture entrapped in void spaces of the roofing tends to expand due to vapor pressure developed therein, the maximum theoretical pressure that may be developed at 170° F. being, we have computed, about 41% greater than normal atmospheric pressure. At lower temperatures, e. g., at 160° F., the pressure differential is less and has been computed to be about 32% greater than normal atmospheric pressure. Bituminous coating materials which have been used heretofore generally have had a softening point of about 210° to 235° F. in commercial practice. Such coating materials become greatly softened or even semi-fluid at summer sun roof surface temperatures such as 170° F. and offer no substantial resistance to the vapor pressure of entrapped moisture and consequently blistering results. While fillers have been used heretofore in bituminous coating materials, the bitumen used when the filler is employed is usually of a somewhat lower softening point than 210° to 235° F. so as to compensate for any effect the filler might have on the softening point of bitumen and so that the softening point of the bituminous mixture will still be about 220° to 240° F. and spreadable at about 300° to 375° F. In this case also, bad blistering may occur if any moisture becomes entrapped in the roofing. Moreover, when fillers have been used heretofore the fillers have not had the suitable character to inhibit the effect of the filler of absorbing water into the coating and thereby causing blistering in the coating or layer itself.

When reference is made herein to a bituminous material or coating or layer, reference is made to a material or coating or layer comprising bitumen either with or without filler. The term "bitumen" is used in referring to asphalt, pitch, tar and the like which have not been mixed with filler material.

The blister barrier coating or layer of roofing embodying this invention does not become weak and fluid at summer sun temperatures as is the case with ordinary bituminous roofing coatings, but, due to its greatly reduced thermal conductivity, remains sufficiently strong and highly viscous at such temperatures so as to enable it to resist the vapor pressure generated by entrapped moisture when subjected to summer sun roof surface temperatures and at such temperatures does not become deformed or stretched so as to result in blister formation. It is this property of the coating or layer that is regarded as being of substantial mental in affording the high blister resistance of roofings embodying this invention.

For bituminous roofing embodying this invention which is coated with a bituminous coating for weather exposure so that the coating weighs about 10 to 25 pounds per 100 square feet, it is desirable that the viscosity of the coating be at least about  $4 \times 10^6$  poises at 170° F. to be highly blister-resistant at summer sun roof surface temperatures as high as this; and for roofing to be sold in all parts of the United States it is preferable that the coating be of the high viscosity mentioned. The viscosity of the roofing de-

scribed in the foregoing example is considerably higher than  $4 \times 10^9$  poises, namely, about  $26.5 \times 10^9$  poises at  $170^\circ \text{F}$ . and is well able to withstand extremely severe conditions of summer sun heat and moisture without blistering. For withstanding extremely severe conditions a viscosity of at least about  $20 \times 10^9$  poises at  $170^\circ \text{F}$ . is preferred, although, in the practice of this invention, the special composition can be made so as to have a considerably higher viscosity than  $20 \times 10^9$  poises at  $170^\circ \text{F}$ . The method used by us in determining the viscosity of the coating material was a modification of the "alternating stress" method described by R. A. Traxler and H. E. Schwyer in A. S. T. M. Proceedings, vol. 36 (1936), pages 518-528, the viscosity value being calculated from the first displacement of the bituminous specimen in the extrusion tube.

Another factor in connection with the coating above described that is material in imparting remarkably high blister resistance, is that the coating itself is substantially non-water absorptive. Thus the coating is adapted for direct weather exposure as the surface is so impervious to water that the interior or body portion of the coating is substantially sealed from penetration of moisture. For this reason, the coating does not become weakened by absorbed moisture so as to lessen the capacity of the filler to reinforce the coating and so as to create blister sources intermediate the thickness of the coating, particularly about the filler particles. Asbestos dust or other finely-divided short chrysotile asbestos is especially desirable for use as a filler for blending with bituminous coating material to reinforce and increase the viscosity thereof according to this invention because of its high and preferential affinity for bitumen rather than for water even at high temperatures and its capacity to afford a substantially non-water absorptive coating or layer.

A convenient test which we have devised for ascertaining whether or not a coating composition is adequately non-water absorptive and resistant to moisture, is to apply the coating composition to a non-porous base sheet such as metal, e. g., aluminum, and then immerse the sheet with the coating adherent thereto in water at about  $185^\circ \text{F}$ . If the coating composition when applied in a layer substantially .020 inch in thickness to the non-porous sheet can be immersed in the water at  $185^\circ \text{F}$ . for about 10 hours or longer without exhibiting small blisters or pockmarks, the coating composition is very highly resistant to water at such temperature and is regarded as "non-water absorptive" as this term is used herein. Coating compositions having such high resistance to water at  $185^\circ \text{F}$ . can readily be made according to this invention and are preferred. If the coating composition under the described test conditions is resistant to water at  $185^\circ \text{F}$ . for about 2 hours or more without exhibiting small blisters or pockmarks, the coating material still has very markedly improved resistance to water at  $185^\circ \text{F}$ . as compared with coating compositions for roofings heretofore made. When it is said herein that the material of a coating composition is resistant to water at  $185^\circ \text{F}$ . it is to be understood that resistance to water under the described test conditions is intended. Moreover, if the coating composition contains a filler, this same test is indicative of whether or not the filler has a preferential affinity for water as compared with its affinity for the bitumen in the composition, and the filler is to be regarded as having a preferential affinity for bitumen in the

composition as against the water at  $185^\circ \text{F}$ . if the bituminous coating composition in question is capable under the described test conditions of remaining substantially free of blisters or pockmarks for 2 hours or longer, for, if the filler had a preferential affinity for the water at such temperature as against the bitumen similarly heated, water would be carried into the coating and blisters and pockmarks would be produced under the conditions of the test.

When it is desired to test the water resistance and non-water absorptiveness of a layer of coating composition of any given thickness desired or utilized in a roofing embodying this invention, the test may be made in an analogous way, namely, by applying a layer of the composition and of the thickness in question to a non-porous sheet such as metal, e. g., aluminum, and immersing the sheet with the coating adherent thereto in water at about  $185^\circ \text{F}$ . The layer is regarded as adequately resistant to water at  $185^\circ \text{F}$ . if it can be immersed in the water at such temperature for 2 hours or longer, without developing blisters or pockmarks, and the layer has preferred non-water absorptiveness and water resistance if it can be immersed in the water for 10 hours without developing blisters or pockmarks. When reference is made to a bituminous coating or layer having resistance to water at  $185^\circ \text{F}$ . it is to be understood that resistance of the coating or layer under the described test conditions is intended.

The coating material was likewise tested for the purpose of ascertaining its stress-strain characteristics both at normal temperatures ( $77^\circ \text{F}$ .) and at elevated temperatures corresponding approximately to maximum roof surface temperatures ( $170^\circ \text{F}$ .). In carrying out the test a specimen 1 cm. x 1 cm. x 3 cm. (between the clips of the testing machine) was used and the test was carried out on a standard ductility testing machine for bituminous materials (A. S. T. M. Standard D113-39) in combination with an indicating scale balance for measuring the stress. The tension head of the ductility machine moved at a rate of 5 cm. per minute and the specimen was kept immersed in water maintained at the specified temperature throughout the test. The tensile strength value was obtained directly from the scale reading at break in grams per sq. cm. When "tensile strength" is referred to herein the tensile strength as thus determined is intended. The ultimate elongation at rupture of the specimen was indicated on an autographic record and calculated in percent elongation. When "percent elongation" is referred to herein the percent elongation as thus determined is intended. The total work done in stressing the specimen to rupture was derived by plotting the percent elongation at each increment of stress until the breaking point was reached to obtain the stress-strain curve of the specimen and then determining from the area between the stress-strain curve and the elongation axis of the graph the work done in gm.-cm. per cu. cm. Where "total work done at rupture" or, more briefly "work capacitance" is referred to herein the value determined as above described is intended.

Referring to the foregoing example of the practice of this invention wherein 40% of asbestos dust was mixed with asphalt, the asphalt per se had a softening point of  $231^\circ \text{F}$ . The viscosity of this asphalt at  $170^\circ \text{F}$ . was  $26 \times 10^9$  poises. In heating such asphalt from  $77^\circ \text{F}$ . to  $170^\circ \text{F}$ . its tensile strength dropped enormously, namely, from 3400 gms. per sq. cm. to 15 gms. per sq. cm.

The total work done to rupture the asphalt sample dropped from 2064 gm.-cm. per cu. cm. at 77° F. to 9.5 gm.-cm. per cu. cm. at 170° F. When 40% of asbestos dust was mixed with the asphalt the tensile strength at 77° F. was increased 1 e., to 7100 gms. per sq. cm. but what is most significant is that tensile strength at 170° F. was increased by the presence of the asbestos dust about 24 times, namely to 386 gms. per sq. cm. Similarly the total amount of work done to effect rupture at 170° F. was increased to 153 gm.-cm. per cu. cm. Thus it is apparent that while the asphalt itself had no appreciable strength or toughness at temperatures such as 170° F. the presence of asbestos dust in an amount such as 40% afforded for the blister barrier coating a material retaining very considerable strength and toughness at temperatures such as 170° F. so as to resist blister occurrence therein.

While very high blister resistance can be achieved using about 40% by weight of asbestos dust as in the example given above, high blister resistance can be attained using lesser amounts of asbestos dust. Thus, when 30% by weight of asbestos dust is employed in the same asphalt the coating composition at 170° F. has a viscosity of  $4.97 \times 10^4$  poises and a tensile strength of 190 gms. per sq. cm. The work to produce rupture at 170° F. is 89 gm.-cm. per cu. cm. The fire resistance of the coating composition is correspondingly reduced, and especially from the point of view of fire resistance use of only 30% of asbestos is a non-preferred embodiment of this invention. Still lesser amounts of asbestos dust such as about 25% by weight are somewhat efficacious from the point of view of blister resistance in connection with a bitumen of the character referred to but do not afford desirably great fire resistance.

For preferred results the tensile strength at 170° F. of the bituminous material for the blister barrier should be at least about 150 gms. per sq. cm. and the work capacitance of the material at the same temperature should be at least about 70 gm.-cm. per cu. cm.

Another advantage of the embodiment of this invention which has been described above is its increased fire retardant character as compared with asphalt roofings heretofore made. Ordinary asphalt felt roofings have certain fire retardant qualities recognized by Underwriters' Laboratories, Inc., which authorize the use on them of their class "C" designation provided the roofing is made to certain specified standards as to materials; construction, and demonstrated ability to pass the class "C" burning brand and flame exposure tests. We do not know of any bituminous prepared roofing or shingle heretofore made on a felt base of organic fiber, irrespective of the weight of the roofing material or the number of layers or piles that are applied to the roof deck that will successfully pass the class "B" or class "A" fire retardant tests of Underwriters' Laboratories, Inc., which tests are reserved for roofing constructions which are able to withstand much more severe fire exposure. We have found that when a roofing such as that described hereinafter, e. g., in the form of shingles and made on an ordinary organic fiber base felt and containing about 40% of asbestos dust in the high viscosity coating, is so highly fire retardant that a three layer covering for a combustible roof deck will successfully withstand exposure to the Underwriters' Laboratories class "A" fire retardant tests. The class 75

"A" fire retardant tests are the most severe fire tests used by the Underwriters' Laboratories. We have found further, that a two-layer (double coverage) application of our improved roofing will successfully pass the Underwriters' Laboratories class "B" fire retardant test.

The fire retardant properties of the new roofing of our invention are principally due to the fact that the blister barrier coating when exposed to direct flame is very highly resistant, even when on a roof deck having approximately a 30% incline, to flowing that leaves the organic felt base substantially unprotected, as is the case with the coating of bituminous roofings of the type heretofore made. Moreover, the special type of coating burns less readily and chars very slowly, and after it does become charred remains in place as a strong, firm and coherent, but highly porous, mass of carbonaceous ash that has a special thermal insulating effect and that does not develop gaps or large cracks permitting the flame to excessively heat an underlying combustible roof deck. Moreover, the coating in itself is so resistant to burning that any spread of flame is prevented and the bituminous coating material ceases to burn after the flame has been removed from the roofing.

A more detailed description of the fire resistance of the roofing follows in connection with typical roofings and roofing structures shown in the accompanying drawings, wherein

Fig. 1 is a plan view of prepared roofing embodying our invention in the form of an individual shingle of simple rectangular shape;

Fig. 2 is a plan view of prepared roofing embodying our invention in the form of an individual shingle of a preferred shape;

Fig. 3 is a plan view of prepared roofing embodying our invention in the form of a shingle strip;

Fig. 4 is a fragmentary cross sectional view on an enlarged scale of prepared roofing embodying our invention;

Fig. 5 is a sectional elevation of an inclined shingle roof embodying our invention without showing the component layers of the individual shingles;

Fig. 6 is an enlarged sectional elevational view of a portion of the roof shown in Fig. 4, as installed;

Fig. 7 is similar to Fig. 5 except that the roofing is illustrated as it appears after exposure to flame temperatures;

Fig. 8 is a plan view of one form of prepared sheet roofing embodying our invention adapted to be installed in roll form;

Fig. 9 is a sectional elevation of a portion of an inclined roof deck with the roofing material of Fig. 7 installed thereon, the roofing material being shown with exaggerated thickness for clarity;

Fig. 10 is a plan view of a typical built-up roof embodying our invention, with parts of the different layers broken away;

Fig. 11 is a sectional elevation on an enlarged scale of a portion of the built-up roof shown in Fig. 10 taken on the line 11-11;

Fig. 12 is a plan view of a special type of built-up roof embodying our invention in which is employed a pre-fabricated granular surface cap sheet as the weather-exposed surface layer, parts of the different layers being broken away;

Fig. 13 is a sectional elevation on an enlarged

scale of a portion of the built-up roof shown in Fig. 12;

Fig. 14 is a fragmentary sectional elevation of precasted felt embodying our invention used in fabricating the built-up roof shown in Figs. 12 and 13;

Fig. 15 is a side elevation partly in section of a fire testing device for testing roofing material; Fig. 16 is a plan view partly in section of the fire testing device;

Fig. 17 is a front elevation of a test panel assembly used in the fire testing device;

Fig. 18 is a side sectional elevation of the fire test panel assembly;

Fig. 19 is a plan view of the fire test panel assembly with parts thereof broken away; and

Fig. 20 is a perspective view of the frame and guard plate used in the fire test panel assembly.

The prepared roofing material may be cut into individual shingles 10 shown in Fig. 1. Preferably the roofing material is cut into individual shingles 10 having a notch or cut-out 49 as shown in Fig. 2 or into strip shingles 11 of the type shown in Fig. 3 having the recesses or slots 50 and the notches or cut-out portions 51. Of course, shingles or the like of other shapes may also be used. In cross section the prepared roofing material appears as in Fig. 4 and comprises a base or foundation sheet 12 of bitumen impregnated roofing felt. Overlying the roofing felt is the coating 14 of special coating material having adherent to the surface thereof and partially embedded therein the slate granules 12. Adherent to the back of the roofing material is a layer 16 of the special bituminous coating material in which is partially embedded and divided sheet 18 of bituminous material 48. Ordinarily some bituminous coating material and dusting material are employed on the back of the roofing material, but these materials are not essential and are sometimes omitted particularly when the roofing material is to be used in constructing built-up roofing.

In Fig. 5 is shown a typical roofing structure in place on a roof deck comprising boards 18 which serve as a support for the shingles, e. g., strip shingles 11 or individual shingles 10. The individual shingles may be 16 inches long overall and laid with a 5 inch exposure thereby affording triple coverage over all parts of the roof deck except for a portion of the spaces between adjacent shingles in each course. The component parts of the individual shingles appear in detail in the enlarged fragmentary view shown in Fig. 6.

A roofing structure of the character of the example above described has extremely high fire resistance. Thus a roofing structure such as that shown in Fig. 6, namely comprising three thicknesses of preformed roofing, will withstand the class "A" fire retardant tests as prescribed by Underwriters' Laboratories, Inc., in their published instructions, (Description of Test Methods to Determine Eligibility of Roofing for Classifications A, B and C, Subject 55, February 1, 1939). These tests are the burning brand test, the flame exposure test and the spread of flame test. In making the class "A" burning brand test the roofing is installed on a roof deck  $3\frac{1}{2}$  feet wide by  $4\frac{1}{2}$  feet long having approximately a 30° incline (5 inches per foot) and a large actively burning brand (weighing about 4 pounds and consisting of a three layer grid of 36 oven dried wood strips  $12'' \times \frac{3}{4}'' \times \frac{3}{4}''$ ) is placed thereon and fanned by a twelve-mile per hour wind so that the brand burns fiercely and with almost a

white heat at the interior of the brand for about one-half hour or more. If the brand burns out without igniting the combustible roof deck (made of 8" white pine boards, finished three sides, spaced about  $\frac{1}{4}$  inch apart) the roofing passes the test.

In the class "A" flame exposure test a deck similar to the deck used in the burning brand test is covered with roofing to be tested and is subjected to a flame produced by a gas burner located below the lower margin of the deck. The flame impinges upon an incombustible apron extending downwardly from the lower margin of the test deck and mushrooms up around the lower margin of the deck and under the influence of a 12-mile per hour wind bathes substantially the full length of the upper surface of the deck covering. The gas flame is applied for periods of two minutes with intervals of two minutes between applications, and if the test is continued for a period of one hour without igniting the combustible roof deck, the roofing passes the test.

The other class "A" fire retardant test is the spread of flame test. This test is carried out under conditions which are the same as those used in the flame exposure test except that the roof deck is 3 times as long (19 ft.) and except that the flame is applied continuously. The flame bathes about three to three and one-half feet of the lower portion of the upper surface of the covering that is applied to the deck and the tendency of any flame resulting from combustion of material in the roof deck covering to spread beyond the area of direct exposure to flame is noted. As long as the flame continues to spread the test is continued until the entire deck is thoroughly ignited. On the other hand if the flame spreads somewhat and then discontinues spreading or recedes, so that it is evident that the flame will not spread further the test is discontinued. If the flame spreads up the deck to a point not more than about six feet from the lower edge of the deck, the roofing passes the class "A" flame spread test.

As mentioned above we do not know of any prepared bituminous roofing heretofore made on a felt base of organic fibers which, irrespective of the weight of the roofing material or the number of layers or plies that are applied to the roof deck, will successfully pass either the class "A" fire retardant tests or will even pass the class "B" fire retardant tests. The class "B" tests are generally similar to the class "A" tests except that for example in the burning brand test the brand is much smaller (consisting of a three layer grid of 18 oven dried wood strips  $3\frac{1}{2}'' \times \frac{3}{4}'' \times 6''$ ) and therefore only one-fourth the size of the class "A" brand) and burns out after a much shorter time interval. In the class "B" flame exposure test, the test is only carried out for one-half hour employing two minute periods of flame application with intervals of two minutes between applications, and in the class "B" flame spread test the flame is permitted to spread up the roofing to a point not more than 8 feet from the lower edge of the deck.

When ordinary bituminous prepared roofing is subjected to the class "A" burning brand test, for example, the burning brand melts the asphalt of the coating composition which, together with any filler included in the coating composition, starts running down the surface of the roof. The melted asphalt also starts to burn and the burning asphalt in running down the roof spreads the fire. The coating material that runs down



the roof leaves the bituminized organic felt base exposed which, being readily combustible, starts burning so that the fire goes through the roofing quite readily and ignites the combustible roof deck in a relatively short time.

By way of contrast our improved roofing made as above described by way of example behaves very differently when subjected to the same burning brand test. The behavior of the roofing material as applied to a roof deck in three layers, e. g., as shown in Figs. 5 and 6, is indicated roughly in Fig. 7. During the exposure to the flame the finely-divided heat resistant material (asbestos dust), due to its surface characteristics including its shape, size and affinity for bitumen, provides a skeletal mat in the special coating 14 and does not flow down the roof and even though the bitumen of the special coating becomes very greatly softened the skeletal mat persists and thereby protects the combustible material underneath. It is also advantageous that in the softened special coating material small bubbles 46 are generated primarily by heat decomposition of the asbestos dust liberating water vapor and that these bubbles expand the coating material somewhat thereby providing increased heat insulation which is effective to shield the deck from the heat of the flame. During the exposure to flame much of the bituminous material carbonizes but the skeletal mat of asbestos dust remains as a firm coherent porous ash having pronounced heat insulation efficiency distributed substantially uniformly over the roof deck. In the roof structure as a whole as illustrated in Fig. 7 the upper layer of special coating material has become expanded and has become somewhat irregular but the partially dehydrated asbestos dust together with residual carbonized bitumen has remained as a porous coherent carbonaceous ash that acts as a protective barrier against the flame. The layer of felt 13 may be carbonized and to a considerable degree may have disappeared leaving air pockets 17 together with some residual carbonized material. The intermediate layer 14 of special coating material is also expanded, the mat of partially dehydrated asbestos dust together with the resulting pyro bitumen remaining in place. The intermediate layer of felt is charred, but is better preserved than the uppermost layer of felt. The bottom layer of special coating material is less severely carbonized than the upper layer and likewise is considerably expanded. The bottom layer of felt is fairly well preserved and the underlying board 18, if charred at all, has not become ignited. The backing layers 15, being of the special coating composition, likewise augment the heat insulating effect of the roof deck covering and decrease the tendency of the bitumen contained therein to flow and to become ignited. It usually takes the burning brand used in making the Underwriters' class "A" burning brand test about 30 to 45 minutes to burn out. During this time the upper surface of the uppermost layer 14 may become red hot but the heat insulation effect of the carbonized residues of the layer 14 (and to lesser degree of the thinner layers 15) is so great that the board 18 does not become ignited and in some cases does not even appear scorched. During the test the effect of the flame on the roofing is confined to the area beneath and closely adjacent to the position of the burning brand and when the brand burns out all

charring of the roofing soon ceases and the roofing cools down. The behavior of the roofing hereinabove described by way of example under the class "A" flame exposure test is generally similar to that above described in connection with the class "A" burning brand test. Such roofing also passes the class "A" flame spread test.

The resistance of the skeletal mat to displacement while the coating material is exposed to flame is due to the surface characteristics of the particles comprised in the asbestos dust. From one aspect the external shape of the particles of the asbestos dust is believed to contribute to the flow resistance of the particles comprised in the coating, inasmuch as while the particles are small they are nevertheless fibrous and tend to form a stable mat for this reason. From another aspect it is believed that the asbestos has the property of stabilizing bituminous material in contact with the surface thereof by an action in the nature of specific adhesion or adsorption and that the stabilization of films of bitumen on the surface of the small particles imparts a matting tendency such that the particles tend to form a stable skeletal mat when the bitumen in the coating is liquified by exposure to high temperatures. In this connection particle size is significant inasmuch as the total surface exercising a stabilizing action is much larger when the particle size is small than when the asbestos is in the form of fibers of considerable length, but excessive subdivision is not desirable. These characteristics are also instrumental in affording a coherence that resists the development of gaps in the coating through which the flame may penetrate during the progress of the test. The expanding of the coating material and development of pores therein are due primarily to the fact that asbestos dust is selected which contains a substantial amount of chemically combined water which under the heat of the flame, becomes liberated forming water vapor which expands the bituminous coating material.

The preformed roofing can of course be laid with a greater proportion of the area thereof exposed and so that the roof covering will be composed of two layers instead of three. Such a roofing is illustrated in Figs. 8 and 9. In Fig. 9 the roofing material is shown as installed on a roof deck with the thickness of the courses exaggerated for clarity. The preformed roofing material consists of a foundation sheet 18 of the bituminous roofing material, coated over a part of its width with a coating 19 of the special coating composition. This material may be made up in rolls about 36 inches wide for example with slightly less than half of the width of the asphalt saturated foundation sheet 18, e. g., 17 inches of the width, coated with the special coating 19 and covered with the mineral granules 20, e. g., slate granules. This type of roofing may be laid on boards 21 of the roof deck using suitable securing means such as nails 47, the slate covered portion being exposed and the balance underlying an adjoining sheet as shown in Fig. 9. Where the preformed sheets overlap they are caused to adhere to each other by a bituminous adhesive material 22 which, preferably, is the special coating composition embodying our invention. The roofing structure above described employing special coating composition embodying our invention which is highly resistant to fire, as the bituminous adhe-

sive 22, will successfully withstand the Underwriters' class "B" tests. If it is merely desired to have the bonding layer of special composition blister resistant with regard to its fire resistance, this layer may be in the form of a high viscosity bituminous composition, e. g., bitumen containing little or no filler, and of the character described more in detail hereinafter.

Of course preformed shingles (either individual or strip shingles) may also be laid in two courses instead of three and when roofing material of the character hereinabove described by way of example is made up into such a double-coverage roof deck covering, the roofing will likewise pass the class "B" tests prescribed by Underwriters' Laboratories, Inc. When a roofing is in the form of preformed shingles or shingle strips, the tabs may if desired be positively held in place by suitable ties, e. g., of metal, or by an adhesive such as a small amount of bituminous material, but this is by no means essential.

It is not necessary in the practice of this invention that the special coating composition be preformed at the factory integral with a foundation sheet composed of bituminized felt or the like inasmuch as the roofing can be fabricated on the job. An example of this type of roofing is shown in Figs. 10 and 11 which illustrate a typical "built-up" roofing. The roofing is shown installed on wood sheathing 22 to which is secured as by suitable nails 23 a single thickness of building paper 24 such as red rosin dry sheet. Two thicknesses of bitumen impregnated felt 26 are secured overlying the building paper by nails 45. A layer of bituminous waterproofing 27 is then applied by mopping it on while in a heat liquefied condition but instead of using ordinary mopping asphalt a special coating composition, e. g., containing about 30% of asbestos rust as described hereinabove, is used and in each layer the mopping asphalt weighs about 25 to 40 pounds per 100 sq. ft. In applying the special waterproofing it should be applied while heated to about 450° F., i. e., at a somewhat higher temperature than the temperature employed in applying ordinary mopping asphalt and to keep the composition uniform an agitator is preferably used in the heater. To facilitate application the special coating composition is preferably mixed on the job mixing preheated asbestos dust with melted bitumen. A layer 28 of bitumen impregnated felt is next deposited on the waterproofing 27 while it is still adhesive. Another layer 29 of the special waterproofing is then installed followed by a top layer 30 of bitumen impregnated felt and a top layer 31 of the special waterproofing composition. Mineral granules 32 may be disposed over the surface of the layer while it is still adhesive and caused to be embedded in the surface.

A built-up roofing of the character above mentioned will be found to be very resistant both to blistering and to fire. If the cap sheet comprises a coating for weather exposure, which coating has the high strength and viscosity above mentioned at summer sun roof surface temperatures, this coating will be highly blister resistant. Moreover, any underlying coating will have sufficient strength and viscosity to prevent blister formation thereon. Built-up roofings heretofore made are susceptible to blistering not only on the weather exposed coating of the cap sheet but also in underlying coatings. Frequently in built-up roofings blisters occur between layers or strata

of felt and over a period of time expand laterally until the blister covers an area of many square inches or even square feet. Any such blister formation is of course very deleterious and results in premature disintegration of the roof covering. According to the present invention this type of blister formation peculiar to built-up roofings can be minimized. The built-up roofing is likewise fire resistant. Thus the built-up roofing it made using conventional methods and materials would only pass class "C" fire retardant tests whereas the roofing above described will pass the class "A" tests. In order to increase the fire resistance of built-up roofings it has heretofore been the practice to place thereon large amounts of mineral, i. e., as much as 300 to 400 pounds per 100 square feet of roofing area. It has also been proposed to employ asbestos felt in the fabrication of the roofing. According to the present invention high fire resistance is achieved without resort to such expedients and using considerably less felt in the roofing as a whole.

A modified form of built-up roofing is illustrated in Figs. 12, 13 and 14. The roofing is shown installed on wood sheathing 22 over which is secured a single layer of building paper 34. Overlying the building paper 34 is a layer 35 of bitumen impregnated felt which instead of being uncoated as in the embodiment shown in Figs. 10 and 11 may comprise a felt base 35a and a coating 35b (see Fig. 14) the coating being preformed at the factory and composed of the special coating composition. In order to secure the next layer 36 of bitumen impregnated felt (which may be precoated like layer 35) in place a suitable mopping asphalt layer 37 may be employed. The mopping asphalt may be a light application of ordinary mopping asphalt inasmuch as the fire resistance is afforded by the special fire resistant composition that is preformed with the felt sheets 35 and 36. However, it is preferable that the adhesive waterproofing that is applied by mopping be composed of the special fire resistant composition of the character herein described so that the roofing will have maximum fire resistance. In a similar way by a waterproof adhesive layer 38 the cap sheet 39 is placed in place. The cap sheet comprises a coating 41 of the special coating composition preformed therewith to which are adherent mineral granules 40, the cross section of the cap sheet being similar to the section shown in Fig. 4 except that the back coating layer 15 is ordinarily omitted. This type of built-up roofing likewise may be made so as to have extraordinarily high blister resistance and fire resistance. In the case of built-up roofings, such roofings may be improved as to blister resistance according to this invention without necessarily increasing the fire resistance by employing as one or more of the layers 27, 29 and 31 in Fig. 11, and as the layer 35b in the composite sheet 35, and/or as the corresponding layers of composite sheets 36 and 38 in Fig. 13, and/or as the adhesive layers 37 and 38 in Fig. 13, special high viscosity bituminous compositions of the character herein described which are not formulated so as to be highly fire resistant. For example, the special bituminous composition may be a high viscosity bitumen used with little or no filler or may be a bitumen containing a filler such as gas carbon black which does not substantially increase the fire resistance of bituminous compositions with which it is used but is effective to form a high viscosity coating having high blister resistance. The formulation of such spe-

cial compositions will be described more in detail hereinbelow.

Referring further to Fig. 13 the composite sheets 35, 36 and 38 may be preformed so as to carry a substantial layer of special coating composition which is both blister resistant and fire resistant, e. g. coating compositions of the character described in the foregoing example. In order to bond such preformed composite sheets together a bitumen may be used having a viscosity of about  $4 \times 10^5$  poises or higher at  $170^\circ$  F., e. g. a bitumen having a softening point above about  $300^\circ$  F., as the layers 37 and 38. In this way the roofing is made highly blister resistant throughout, and, if the layers 37 and 38 are made relatively thin as compared with the fire resistant layers containing filler, the fire resistant properties of the roofing covering as a whole will not be materially impaired.

We have devised a suitable test for determining the fire retardant properties of bituminous coating material for roofing and its behavior under flame exposure which test consists in directing a flame against an inclined specimen of bituminized felt carrying the coating material to be tested under precisely controlled conditions determined by the construction and operation of the testing apparatus. This test which we have originated is suitable for testing roofing materials without actually carrying out the extensive fire retardant tests prescribed by the Underwriters' Laboratories, Inc. which are on a much larger scale and involve much more labor and expense. The test while not a full substitute for the more elaborate tests prescribed by the Underwriters' Laboratories is capable of giving precise and reproducible results that are indicative of the fire retardant properties of the roofing that would be determinable under the Underwriters' tests and this has been demonstrated in connection with roofing materials of the character described herein. The following is a description of the controlled conditions of the test, reference being made to Figs. 15 to 26 of the drawings:

Samples of material to be tested are made by applying bituminous coating material to an asphalt saturated felt base sheet so that the coating weighs 25 pounds  $\pm$  2 pounds per 100 sq. ft. No granular material or the like is applied to the surface of the coating. The saturated felt base sheet weighs about 30 pounds per 100 sq. ft. and is saturated to the extent of about 175% by weight of the dry felt with asphalt having a softening point of about  $150^\circ$  F. The bituminous coating material is applied by coating the felt in the machine direction of the sheet and the felt is applied to the test deck so that the machine direction of the sheet is parallel to the direction of flame travel.

The test is made in the wind tunnel 53 having a fan 54 at one end and a stack 55 at the other end. The tunnel is made of  $\frac{1}{4}$  inch thickness asbestos-cement lumber and has two windows 56 and 57 therein which can be opened and closed by any suitable means (not shown).

Within the tunnel is the burner and testing deck which are located between two shields 58 and 59 of the asbestos-cement lumber spaced 12  $\frac{1}{2}$  inches apart, and which are rigidly mounted on a steel slab 43. The inclined test deck is indicated generally by the reference character 59 and comprises a lower iron frame-like member 60 having  $\frac{1}{4}$  inch pegs 61 projecting from the face adjacent the upper and lower margins. Between the pegs strips 62 of combustible ma-

terial  $1\frac{1}{4} \times 12 \times \frac{1}{4}$  inches are placed. This combustible material is what is known as "Masonite Quarterboard" and is selected instead of wood because it can be obtained with greater uniformity than can wood. "Masonite Quarterboard" is made from wood fiber by compression of the fiber of a fibrous mass in the presence of its naturally contained lignin binder until it has a density of about 36 pounds per cubic foot. The four center boards are dried at  $180^\circ$  F. in a steam oven for at least 7 days. Two full plies 12 x 12 inches of the roofing 59 to be tested and one-half ply 12 x 6 inches are placed on the combustible deck followed by an L-shaped guard plate 63 which guards the bottom edge of the roofing and of the Masonite. The assembly is held down by an iron frame 64 open at the bottom and held in place by thumb screws 65.

After the test deck has been assembled it is placed on the inclined support 66 which has an opening in the back underneath the combustible material 62 and which has side flanges 68 to protect each side of the test panel. The support 66 comprises a baffle 67 to prevent the flame from licking around behind the test deck. The support which is made of iron is mounted on the steel slab 43 which measures 12 x 40 x 1 inches. The parts for carrying the test deck are also made of iron. An iron bar 69, 12 x 1 x  $\frac{1}{4}$  inches, is placed across the top edge of the deck to protect the combustible material at this point.

In front of the test deck is the burner 70 comprising a  $\frac{3}{4}$  inch outside diameter standard gas iron pipe with 17 holes 0.078 inch in diameter and  $\frac{1}{4}$  inch apart disposed at an angle that is parallel with the plane of the test deck. The burner has one inlet 71 controlled by valve 72 and another inlet 73 controlled by valve 74.

The dimensions of different parts of the testing apparatus shown on the drawings as used by us are as follows:

Inches	Inches
a..... 24	o..... 8
b..... 38	p..... 2
b'..... 6	q..... $\frac{3}{4}$
c..... $18\frac{1}{2}$	r..... 1
d..... 72	s..... 12
e..... $16\frac{1}{2}$	t..... $\frac{7}{8}$
f..... 10	u..... $10\frac{1}{2}$
g..... $12\frac{1}{2}$	v..... 12
h..... 38	w..... 12
i..... 64	x..... 1
j..... 32	y..... 12
k..... 24	y'..... 1
l..... 24	z..... 6
m..... 40	aa..... 12
n..... 27	bb..... 1

In carrying out the test the apparatus is first assembled and the burner is lighted, the valve 72 being opened and adjusted until a pilot flame is produced that is about  $\frac{1}{2}$  inch in length when the fan is operating. The windows 56 and 57 are then closed and the room in which the apparatus is placed is arranged so that there will be relative constant conditions during the test. The temperature and relative humidity of the room may suitably be approximately  $80^\circ$  to  $85^\circ$  F. and 30 to 50% respectively. The fan should generate a wind velocity of about 150-155 feet per minute at the portion of the deck exposed to the flame. The valve 75 is then opened until a flame about 8 to 9 inches long is produced having a temperature of about  $1325-1370^\circ$  F. When the valve 75 is opened to increase the flame a timing device is started.

During the test the upper surface of the test deck can be observed as well as the back composed of combustible Masonite strips. During the test the behavior of the coating material can be noted including possible exposure and combustion of the felt base sheet, the flowing of coating material down the roof deck, etc. When one or more of the Masonite strips of the test deck catch fire and begin to burn the test is concluded. If the Masonite merely carbonizes without breaking into flame the end point has not yet been reached.

In carrying out the test as above described the heat of the flame first softens the bituminous coating material and with ordinary roof coating materials used on prepared roofing products at present on the market the bituminous coating material including any filler disposed therein becomes displaced from the felt base sheet and flows down leaving the base sheet material substantially unprotected with the result that the organic felt material of the roofing in the test panel is consumed and the heat of the flame strikes through so that the combustible panel deck relatively soon becomes ignited. However, when the piles of the asphalt saturated felt base sheet material are coated with special coating composition according to this invention, the test samples behave in a manner corresponding generally with the behavior hereinabove described of roofing material embodying our invention when it is subjected to the Underwriters' Laboratories class "A" burning brand and flame exposure tests. The finely-divided asbestos dust or the like instead of flowing down the inclined test panel when the bitumen in the coating material becomes heated and softened by the flame resists the tendency to flow down the panel and provides a skeletal mat which remains in place as a coherent heat insulating mat-like mass in an amount adapted to effectively protect the underlying combustible test deck from the heat of the flame for a considerable period of time. Some of the bitumen in the coating may flow somewhat but most of it is retained in the skeletal mat which persists in place and resists displacement and remains as a protective covering. Eventually during the test the bitumen becomes hardened by carbonization and contributes to the coherence and toughness of the mat-like protective mass that remains. During the continuance of the test coating material having high fire retardant properties according to this invention continues to protect the combustible deck material from the heat of the flame so that the combustible deck does not ignite even under the very severe conditions of the test for prolonged periods of time, e. g. 40 minutes and upward of two hours or more. The resistance to flow of the finely-divided asbestos dust or the like is due to the surface characteristics thereof which have been discussed more fully hereinabove and is such that a stable coherent skeletal mat is provided which affords heat insulation against the heat of the flame.

The proportion of the finely-divided filler material that is used in the coating composition and the properties of the bitumen of the character herein mentioned with which it is mixed may vary, but in any event the proportion of finely-divided filler to bitumen should not be so great that the composition cannot be spread in a heat plasticized condition in a substantially uniform layer weighing about 30 to 60 pounds per 100 sq. ft. on sheet material by means of a doctor using conventional coating machinery of the type used

In the manufacture of prepared asphalt roofings. Any bituminous composition which can be so spread is referred to herein for the sake of brevity as a "spreadable" coating composition. As mentioned more in detail elsewhere herein, the amount of bitumen binder should not be less than about 45% by weight of the bituminous coating composition inasmuch as coating compositions containing lesser quantities of bitumen do not have the desired spreadability. On the other hand, in the case of certain filler materials coating compositions containing them may be somewhat lacking in desired spreadability, even when the coating composition contains a greater proportion than 45% by weight of the bitumen. If a finely-divided filler material, when used in a coating composition in an amount which does not render the composition non-spreadable, is, due to its surface characteristics, sufficiently resistant to flow to provide a stable skeletal mat that, under the conditions of the test above described, protects the combustible deck from ignition for a period of about 40 minutes or longer, the filler material is regarded as "flow resistant," as this term is used herein, when the bituminous composition in which the finely-divided filler is contained is exposed to flame temperature. In such case the finely-divided filler material has the special property of resisting the tendency to flow down the test panel when the bitumen is melted due to exposure to the flame temperature and of persisting in place to provide the high resistance to flame exposure indicated by the test, and the test which we have devised and described above enables one to readily ascertain this flow resistant property of suitable finely-divided filler materials.

The behavior of roofings embodying this invention may be contrasted with the behavior of the coating material used on ordinary roofings under the test conditions above mentioned. Ordinary asphalt impregnated roofing felt which weighs about 30 pounds per 100 sq. ft. and which is not protected by any waterproofing coating will prevent the combustible deck material from igniting for about 18 minutes. If the felt is coated with ordinary coating asphalt having a softening point of about 230° F., (the coating weighing for example about 25 pounds per 100 sq. ft.) the coating asphalt being itself combustible promotes the combustibility of the felt and the test can be run for only about 13 minutes without igniting the combustible deck. If ordinary filling material such as slate dust is included in the coating material (the coating composition containing for example about 35% of the slate dust) the combustible deck will ignite in about 17 minutes showing that the slate dust is not so effective to afford fire retarding properties. Slate dust is specifically mentioned since slate dust is the filler most extensively used by the industry for the coating material used on bituminous roofings. Notwithstanding the presence of the slate dust the bituminous coating ignites readily and becomes displaced from the felt so that the roofing rapidly disintegrates and permits the combustible deck used in the test to become ignited after only the short period of time mentioned. Limestone dust when used as a filler behaves very similarly to the slate dust. Materials such as white lead, asbestine and pyrophyllite talc likewise have little effectiveness.

It is apparent from the results of the test above mentioned that according to the present invention one can readily double fire resistance as

compared with the fire resistance of ordinary roofings made at the present time and in preferred embodiments of this invention one can increase the fire resistance of roofings many times more.

Hereinafter emphasis has been placed chiefly on the blister resistant and fire resistant properties of the roofings embodying this invention. It may also be mentioned, however, that roofings having these advantages can be made according to this invention without sacrifice of other desired properties and even with resulting improvements in other respects. Thus roofings embodying the special coating material can be manufactured which have adequate pliability at ordinary temperatures. Roofing material made according to the previously described preferred example can be bent 180° in 2 seconds around a 2 centimeter diameter mandrel with the blister barrier coating on the outside without cracking of the coating through to the felt base, the test being made at 77° F. The roofing as above described has the advantage of being somewhat stiffer at normal temperatures than ordinary asphalt roofings heretofore manufactured. While it is possible to achieve very high pliability at normal temperatures in the practice of this invention, one can achieve the advantages of this invention in roofings which have somewhat less pliability at normal temperatures, e. g. 77° F., but which still have sufficient pliability at normal temperatures for most commercial purposes. It is desirable that an asphalt roofing be at least sufficiently pliable to be bent 180° in 2 seconds around a mandrel of 10 centimeters diameter at 77° F. with the coating on the outside, without cracking the coating through to the base on which it is applied and any such coating or layer is referred to herein as "pliable" at ordinary temperatures.

The new roofing of this invention described in the foregoing example is likewise less brittle at lower temperatures such as 50° and 32° F. than roofings heretofore made, and can be applied in cold weather better than such roofings.

The new roofing described in the foregoing example also exhibits an increase in strength and toughness as compared with ordinary roofings of substantially the same weight, the increase being in the order of about 20% when subjected to the Mullen bursting test (with the coating for weather resistance outward) or to conventional tensile strength tests at 77° F.

It may also be mentioned that the new roofing of the previously described example exhibits improved ability to hold the embedded granular surfacing material in the coating layer during weather exposure because of the toughness and strength of the high viscosity bituminous coating resulting in decreased tendency of the surfacing material to loosen or shed under weathering conditions as compared with ordinary bituminous roofings.

In addition to the foregoing properties and characteristics of roofings embodying this invention, mention also should be made of the highly important fact that the new roofing can be made on conventional roofing machinery and that the special coating can be applied by a conventional roofing coating operation. It is of great practical importance that the special composition comprises asbestos dust which does not interfere with the spreading of the special coating composition uniformly on the felt with the

asbestos dust distributed as a uniform mat-like mass therein.

In the manufacture of roofing according to the present invention utilizing the method and apparatus described in our Patents Nos. 2,105,531 and 2,159,586 it is not essential that the felt base be sealed with the special coating composition. For example, the felt base may first be impregnated with a bituminous saturant then sealed in a devoided condition with ordinary coating asphalt having a softening point of about 220° to about 240° F. Thereafter the filled and sealed web could have a coating of the special coating composition applied to the surface thereof and thereby afford the advantages of a blister resistant coating or barrier for the roofing which coating or barrier preferably is also fire resistant. Moreover, instead of devoiding the fibrous web base to as low as about .5% as described above, a void content of the fibrous web base and of the roofing of less than about 2% affords good resistance of the fibrous web base to penetration by moisture and to the deleterious action of moisture and is regarded as being substantially non-porous.

While it is preferable that the blister barrier coating or layer be carried by a felt base that has been filled and sealed in the manner described in our Patents Nos. 2,105,531 and 2,159,586 this is not essential. For example, roofing that is manufactured merely by first impregnating the roofing felt with a bituminous saturant having a softening point of about 110° to about 160° F. and then applying the special coating material having high viscosity at summer sun roof surface temperatures according to the present invention to the felt which is still porous, e. g. containing about 10 to 20% of voids, exhibits greatly improved resistance to blistering as compared with roofings utilizing ordinary bituminous coating material applied to a similar felt base because the special coating has such high strength and resistance to deformation that vapor pressures generated by moisture entrapped in the porous felt base by summer sun temperatures do not deform the coating and form blisters in or under the coating layer. The roofing as thus prepared has somewhat less resistance to the boiling water test than the roofings described in the preceding examples, but this is due primarily to failure of the roofing felt at the back of the roofing rather than to blister formation in the special high viscosity coating. Many roofings fail on weather exposure due to disintegration of the felt at the back of the roofing and for this reason it is preferable to utilize the method and apparatus of Patents Nos. 2,105,531 and 2,159,586 in the manufacture of roofing according to the present invention so that the surface coating will be blister-resistant and at the same time the felt base will not deteriorate prematurely and will have as long a life as the blister-resistant coating. A roofing made without utilizing our prior patents will have the fire resistance, pliability, strength and toughness that are incident to preferred embodiments of this invention above described.

While it is preferable to employ a filler in the blister barrier bituminous coating or layer it is not absolutely essential to do so while still obtaining improved blister resistance according to this invention. Thus a high viscosity coating can be used which merely consists of bitumen which has been refined and oxidized to a much higher softening point than the softening point of bitumens heretofore used in the manufacture of

prepared asphalt roofings. In such case it is preferable that the softening point be as high as about 300° F. (ball-and-ring) to impart high blister resistance and to afford a roofing that will withstand summer sun roof temperatures as high as about 170° F. The viscosity of such bituminous material is about  $30 \times 10^5$  poises at 170° F. For withstanding summer sun temperatures somewhat below about 170° F. such as are encountered in the northern half of the United States or for affording somewhat less blister resistance at higher summer sun temperatures, a coating of bitumen having a viscosity of about  $4.0 \times 10^5$  poises at 170° F. may be used.

A roofing which comprises a blister barrier coating consisting substantially entirely of bitumen having high viscosity at summer sun roof surface temperatures is not as satisfactory as the embodiment of this invention containing asbestos dust as a filler, inasmuch as while the coating has high resistance to blistering it has considerably less stiffness and toughness at temperatures such as 77° F. and is less pliable and more brittle at lower temperatures such as 32° F. The roofing also has shorter weathering life in use. Moreover, the fire resistance is only slightly, if any, greater than that of bituminous roofings heretofore manufactured because the high viscosity bituminous coating is not stable against melting and flowing at flame temperatures. When the blister barrier coating is composed substantially entirely of high viscosity bitumen, it is preferable to use an air-blown asphalt that has been brought to the proper viscosity by heat and oxidation and to use bitumens derived from Mid-Continent petroleum in order to achieve as high resistance to weathering as possible, but such bitumens have the disadvantage of being the "staining" type that causes discoloration of many but not all mineral surfacing granules that are embedded in the coating asphalt layer.

It is possible in the practice of this invention to incorporate a filler such as asbestos dust in a bitumen such as bitumen having a softening point greater than about 275° F. or even greater than about 300° F. and thereby obtain greater toughness, pliability, fire resistance and some increased resistance to weather exposure. It is not necessary, however, when a filler such as asbestos dust is used to employ a bitumen that has such a high softening point (before incorporation of filler) in order to obtain a high viscosity coating that is resistant to blistering. As mentioned above by using a bitumen that has a softening point of about 220° F. to about 240° F. and using asbestos dust therewith so that there is about 30% of the asbestos dust in the coating as a whole, the viscosity of the coating can be raised to  $4 \times 10^5$  poises or higher at 170° F. and to a softening point of about 320° F. By thus utilizing a bituminous material having initially a relatively low softening point together with sufficient filler such as asbestos dust to afford a high viscosity, high softening point coating material optimum conditions are obtained, namely, blister resistance combined with high pliability at ordinary and at low atmospheric temperatures, long weathering properties and fire resistance.

Somewhat more generally, it is preferable to employ in the practice of this invention a bitumen having a softening point below about 275° F. and preferably below about 240° F., e. g., about 200° F. to about 240° F., together with a filler that is of such character and amount as to raise the softening point to above about 300° F., although

the softening point of the bitumen does not have to be quite as high when a filler is used as when a filler is not used. It is usually not desirable to vary the softening point of the bituminous material above about 350° F. (either containing or not containing a filler) inasmuch as the coating material becomes undesirably stiff and brittle at ordinary temperatures and is difficult to apply by a coating operation in a heat-plasticized semi-fluid state.

Preferably the bitumen that is used is that which is obtained by air-blowing a residual asphalt flux from the refining of Mid-Continent petroleum or a flux of similar characteristics, such as the residuum from Illinois crude, to a softening point of about 220° F. to about 240° F. inasmuch as such an asphaltic material affords a blister barrier coating having very high resistance to weathering, combined with good pliability. Use of soft oily bitumens having a softening point below about 160° F. is not recommended. Air-blown asphalts are preferable to steam distilled and straight run residual asphalts because of their lower temperature susceptibility. Other bituminous and asphaltic materials may also be used such as asphalt produced from the refining of Mexican, Venezuelan and Colombian crudes. Moreover, various pitches, coal tar and the like may be used in the practice of this invention, and are classified herein as bitumens, the term bitumen being used in a broad sense and as including binder materials that contain at least to a predominant extent thermoplastic binder materials of the character aforesaid. It is not essential when a filler is used that the softening point of the bituminous material be elevated to above 300° F. if the viscosity and tensile strength are increased sufficiently to inhibit blistering. It is viscosity and strength at summer sun roof surface temperatures that are of paramount importance. As aforesaid, however, the use of bituminous compositions for the special coating having a softening point above 300° F. is to be preferred.

In addition to asbestos dust there are a number of other finely-divided fibrous asbestos filler materials, that may be used. There are various other materials such as those referred to as "asbestos powder" and "milled asbestos tailings" which are highly desirable. Also available are various types of chrysotile asbestos refuse or shorts produced in the milling and grading operations. Another fine asbestos filler which may be used is that which is known as "asbestos air chamber floats" and which is recovered by air flotation from the milling and screen grading of asbestos fiber. This material is not quite as desirable as asbestos dust. These asbestos fibrous materials are all of fine particle size and very short fiber length. While the various types and grades above mentioned vary to some extent in grading and fineness of subdivision they are all such that a major proportion will pass a 48-mesh standard testing sieve. Also with the exception of the asbestos air chamber floats, which are somewhat less desirable, they all contain at least about 50% by weight of material which will pass a 48-mesh sieve, at least 35% by weight of material which will pass a 100-mesh sieve, at least 20% of material that will pass a 200-mesh sieve, and at least 15% of material that will pass a 325-mesh sieve. While these materials are very finely divided and of short fiber length they are predominantly fibrous in character.

A filler material which is fibrous in character

is preferred in the practice of this invention inasmuch as fibrous fillers have greater effect in the amount used in increasing the toughness, pliability and fire-resistance of the coating. Moreover, it is preferable to employ a graded fiber mixture of which at least 25% is retained on a 100-mesh sieve, at least about 35% passes a 100-mesh sieve and at least 20% passes a 200-mesh sieve. For combined blister resistance and fire resistance it is preferable that about 20% by weight of the fiber be retained on a 48-mesh sieve and that about 40% by weight be retained on a 150-mesh sieve.

While finely-divided asbestos should be used, it is possible to get the material excessively fine. Thus if asbestos is screened and only the residue is used, which residue will pass a 200-mesh testing sieve, both the blister resistant and fire resistant characteristics of the coating material are reduced. This is believed to be due to the fact that such extremely fine material lacks the capacity to impart strength to the coating sufficient to make the coating highly resistant to blistering and also lacks the capacity to form a skeletal mat-like mass which is adequately stable and resistant to displacement when the coating is subjected to flame temperatures. For this reason when finely-divided asbestos is used, the coating composition should preferably contain at least about 10% of asbestos that is retained on a 100-mesh testing sieve.

On the other hand, the asbestos that is used should not comprise an excessive amount of fiber having considerable length. The grades of asbestos classified as "fiber" according to the Quebec Asbestos Producers Association are unsuitable for use in our invention because their average length is excessive. Such grades of asbestos which contain fibers of very substantial length (and which are desirable for this reason for a number of other commercial purposes such as reinforcing insulation, manufacture of asbestos paper, etc.) do not lend themselves to the improved roofing of this invention. The fibers tend to become entangled in clumps or clots which result in non-uniform distribution of the asbestos in the coating composition and which render it substantially impossible in a manufacturing operation to apply the coating material without producing a product of non-uniform thickness and having an unsatisfactory irregular surface texture and therefore commercially unacceptable. These irregularities likewise impair the weather resistance and durability of the roofing on exposure and also impair the fire resistance of the roofing. For these reasons the coating material preferably should not contain more than about 10% by weight of the total asbestos content that is retained on a 14-mesh sieve.

Considering then the matter of fiber size, it is preferable in achieving blister resistance and in achieving fire resistance to employ fiber which passes a 14-mesh testing sieve and a substantial portion of which is retained on a 150-mesh sieve, a major proportion being retained on a 200-mesh sieve. A somewhat wider range is, however, permissible, namely, passing a 10-mesh testing sieve with a major proportion retained on a 200-mesh testing sieve. When it is stated herein in the claims that the bituminous composition contains a minimum percentage of fiber or other filler which passes a 14-mesh (or a 10-mesh) testing sieve and a major proportion (over 50%) by weight of which is retained on a 200-mesh testing sieve, reference is made to the defined filler material as being of particular effective-

ness for use according to this invention but without necessarily excluding the presence of additional fiber or other filler that is outside of this definition. Thus, in the example of this invention that was given hereinabove, the bituminous composition of the waterproofing layer contained 40% by weight of asbestos dust, all of which passes either a 14-mesh (or a 10-mesh) testing sieve and substantially 50% by weight of which is retained on a 200-mesh testing sieve. Such a bituminous composition is to be understood as containing 40% by weight of filler which passes a 14-mesh (or a 10-mesh) testing sieve and a major proportion of which is retained on a 200-mesh testing sieve, and a bituminous composition is to be understood as containing this same percentage of such effective filler material, if, in addition to this percentage of effective filler material, there may be present some additional filler that passes a 200-mesh testing sieve or some coarse material that is retained on a 10- or on a 14-mesh testing sieve. More concretely, and merely by way of example, if there is available a supply of asbestos dust all of which passes a 14-mesh testing sieve and containing 50-51% by weight of particles retained on a 200-mesh testing sieve, and a bituminous composition is formulated using 40% by weight of the composition of this asbestos dust, the resulting composition obviously contains 40% by weight of finely-divided mineral filler which passes a 14-mesh testing sieve and a major proportion (over 50%) by weight of which is retained on a 200-mesh testing sieve regardless of the balance of the composition which may consist entirely of asphalt or may consist of asphalt together with some other filler such as asbestos retained on a 14-mesh testing sieve and constituting say, 3% by weight of the composition, and/or such as asbestos powder, hydrated Portland cement powder, or the like, all passing a 200-mesh testing sieve and constituting say, 5% by weight of the composition. In a composition of the proportions just mentioned, the 3% by weight of asbestos that is retained on a 14-mesh testing sieve and the 5% by weight of additional filler all passing a 200-mesh testing sieve merely constitute surplus filler in the composition which as aforesaid, contains 40% by weight of filler which passes a 14-mesh testing sieve and a major proportion (over 50%) by weight of which is retained on a 200-mesh testing sieve. The foregoing applies to other fillers and filler mixtures and to percentages of filler content other than 40% that are mentioned herein.

The use of finely-divided asbestos filler material is preferred not only because of its pronounced effect in increasing the viscosity and strength of bituminous material with which it is mixed, but also because of its pronounced ability to provide a bituminous composition that when disposed as a coating for roofing is extremely highly resistant to absorption of moisture. When finely-divided fibrous asbestos dust is used as the filler in a bituminous coating the entire thickness of the material is substantially completely sealed against penetration by moisture and is therefore highly resistant to the development of blisters intermediate the thickness thereof. For example, if a coating material consisting of asphalt and about 25% by weight of reinforcing fine asbestos is coated on metal, the coated metal can be immersed in water at 185° F. for 10 hours or longer without exhibiting any tendency for the asphalt coating to blister.

Some fillers are not suitable in the practice of this invention. Vegetable fillers such as wood, cotton, cellulose and scrap roofing felt containing organic fibers are generally undesirable. For example, wood flour which is highly absorptive and tends to swell when moist, tends to permit water to become absorbed thereby even though it is dispersed in bitumen with the result that a bituminous coating containing wood flour as a filler when coated on a metal base will not withstand the immersion test in water at 185° F. but generates blisters intermediate the thickness thereof. For the same reason highly water absorptive mineral fillers, e. g. bentonite, are relatively undesirable. Those fillers that are porous, such as diatomaceous earth or which have high surface affinity for water such as calcium carbonate and pulverized silica (quartz), are also undesirable particularly when used with a bituminous material of relatively low softening point, e. g., a softening point of about 240° F. Water-soluble filler materials are undesirable. In general, it is preferable to employ a non-porous water-insoluble mineral filler that has a preferential affinity for bituminous material rather than for water in order to provide a non-water absorbent bituminous composition.

Fillers such as limestone flour and slate flour do not appear to have the capacity to increase viscosity and tensile strength to desired extent. Limestone flour and slate flour are prepared by pulverizing natural limestone or slate. Representative specimens of these filler materials containing over 80% of particles passing a 100-mesh testing sieve were mixed with asphalt having a softening point of 231° F. Even when as much as 40% of these fillers were used the viscosity at 170° F. was only  $5 \times 10^4$  poises and additional quantities of these filler materials within the limits of workable consistency of the composition did not materially increase the viscosity at 170° F. of the bituminous material. Thus 50% of limestone flour gave a viscosity of only  $.81 \times 10^4$  poises at 170° F. The tensile strength at 170° F. using 40% of limestone flour and slate flour was 60 gms. per sq. cm. and 75 gms. per sq. cm., respectively, and the work capacitance at 170° F. was slightly less than 22 gms.-cm. per sq. cm. for each. It is therefore apparent that limestone flour and slate flour even though very finely divided lack the effectiveness of the asbestos dust to afford high viscosity and high strength whereby blister resistance can be achieved. Moreover, the materials containing limestone flour or slate flour could not, when coated .020 inch thick on metal, be immersed in water at 185° F. for half to three quarters of an hour without blistering, indicating excessively low resistance to water absorption.

Of the filler materials that may be used in the practice of this invention chrysotile asbestos shorts or dust are preferred. This material affords a coating which is highly resistant to moisture penetration and is adapted to increase the strength of coating compositions so that they are substantially impervious to blistering at summer sun temperatures. In the second place the material has the capacity to form a stable and coherent skeletal mat that is highly flow resistant at flame temperatures. In the third place, this material contains water of constitution liberatable at temperatures adjacent flame temperatures which, by forming pores when the composition is subjected to flame temperatures, greatly increases the thermal insulating effect of the

composition when subjected to flame. Asbestos fibers of other types when of the fineness of size or grading above specified and which contain water of constitution liberatable at flame temperatures, are preferable for use in our invention for the same reason. Another fibrous material, Canadian picrolite, which also has these properties, comes within the meaning of the term asbestos as used herein and may also be used when of the fineness of subdivision hereinabove described. There are other fibrous minerals of the asbestos group which have low liberatable combined water and therefore do not liberate water vapor at flame temperature, but which are suitable, in the finely-divided condition above described, from the standpoint of blister resistance and forming a flow resistant and coherent skeletal mat that is fire resistant. Such additional abestiform mineral fibers include crocidolite, amosite, anthophyllite, tremolite and actinolite.

Another material which is excellent from the point of view of affording blister resistance and also fire resistance is finely ground asbestos cement scrap. Asbestos cement roofings usually contain about 30% to 35% by weight of asbestos fiber and 65% to 80% by weight of hydrated Portland cement. Heretofore such scrap material has been regarded as an unavoidable waste of no commercial value. However, by subjecting such scrap material to a disintegrator such as a hammer mill, until it at least passes a 10-mesh testing sieve and preferably passes a 14-mesh testing sieve and until about 50% by weight passes a 48-mesh testing sieve, a material is afforded which when incorporated, e. g. to the extent of about 30% to 45% by weight of the coating composition affords a roofing which is very highly blister resistant and is likewise very highly fire resistant. Preferably a major proportion of the ground asbestos-cement scrap is retained on a 200-mesh testing sieve. Thus if a coating composition containing for example 35% of the finely ground asbestos-cement scrap and asphalt having a softening point of about 230° F. is applied to an aluminum panel, the panel can be inserted in water at 185° F. or even in boiling water, for 24 hours and longer without exhibiting any blistering tendency, thereby indicating its very high blister resistance and resistance to moisture penetration. The effectiveness of asbestos-cement scrap is due partly to the fibrous character of the particles and to the fact that the fibers carry nodules of the cement thereby affording a highly stable mat in the coating which renders the coating both fire resistant and blister resistant. Moreover, both the asbestos and the hydrated cement have an affinity for asphalt which is preferential to water even at elevated temperatures and which stabilizes asphalt films on the surface thereof so as to impart flow resistance at flame temperatures. Likewise both the asbestos and the Portland cement give off water of constitution at elevated temperatures which contributes to fire resistance as has been mentioned more in detail elsewhere herein. Hydrated Portland cement contains about 15% to about 20% by weight of water of constitution which is liberatable at temperatures somewhat below flame temperatures, namely, at temperatures above about 800° F.

In addition to asbestos-cement roofing scrap other sources of mixed asbestos and hydrated Portland cement in the finely-divided form above described may be availed of. Mixtures of abestiform mineral fibers with hydrated Portland ce-



ment whether or not in the form of ground scrap are referred to herein as "asbestos-cement."

Hydrated (indurated) Portland cement is also useful by itself in the practice of this invention and should preferably be produced, e. g. by pulverization, so as to pass a 48-mesh testing sieve. Particles which are retained on a 14-mesh testing sieve are undesirable. Hydrated Portland cement of such fine particle size will by itself serve to impart quite high blister resistance but not as high blister resistance as ground asbestos-cement scrap or asbestos dust when corresponding amounts of mineral filler are used in a coating composition. Hydrated Portland cement also imparts some fire-resistance to roofings but not as high fire resistance as ground asbestos cement scrap or asbestos dust.

Another material which is analogous to hydrated Portland cement in the extent to which it imparts both blister resistance and fire resistance to the special coating compositions is powdered serpentine rock. This material is especially effective when produced so as to pass a 48-mesh testing sieve and be retained on a 200-mesh testing sieve and should pass a 14-mesh testing sieve. While hydrated Portland cement and powdered serpentine rock are not fibrous in character they nevertheless have the capacity to provide a stable and coherent skeletal mat that is flow resistant when the coating material is exposed to flame temperatures. This property is believed to be due primarily to the fact that these materials have the property of stabilizing films of bitumen in contact therewith. Powdered serpentine rock, like hydrated Portland cement, contains water of constitution liberatable adjacent flame temperature and this property also contributes to the fire resistance of bituminous coating compositions containing them. Somewhat more generally the requirement that the coating material comprised by the new roofing provides a protective skeletal mat when the coating is subjected to flame temperatures is the result of surface characteristics of the small heat resistant particles contained in the coating material, i. e., the shape and size thereof and/or interfacial action between the particles and the bitumen which stabilizes bitumen films in contact with the surface of the particles. Any finely-divided heat resistant material, the particles of which have such surface characteristics as to be flow-resistant, may be used to afford a roofing having high fire resistance. By "heat resistant" any material which is sufficiently heat resistant that it will retain structural integrity when subjected to flame temperature while incorporated in a roofing is intended. In this connection asbestos fibers, hydrated Portland cement, picro-lite, etc., which contain water of constitution that may be driven off at temperatures adjacent flame temperatures are regarded as heat resistant.

The finely-divided materials hereinbefore specifically mentioned have utility both because of their capacity to increase blister resistance and because of their capacity to increase fire resistance. In addition there are certain mineral filler materials which, while they do not materially increase the fire resistance of roofing nevertheless can be used so as to produce a coating having such strength, viscosity and water impermeability at summer sun roof surface temperatures as to be highly blister resistant. Gas carbon black produced by pyrolytic decomposition of natural gas by the well known channel process

is a typical example of a filler material of the character referred to. Gas carbon black affords a non-water absorptive high viscosity blister barrier coating. It does not, however, afford a coating that is of as high fire resistance as coatings containing asbestos dust or the like. Gas carbon black is however very effective in imparting high viscosity and strength at 170° F. Thus, a bituminous composition consisting of asphalt having a softening point of 231° F. and 25% of gas carbon black had a viscosity of 40.7×10<sup>3</sup> poises at 170° F. and a tensile strength of 455 gms. per sq. cm. at 170° F. The work capacitance at 170° F. was 173.5 gm.-cm. per cu. cm. When a coating 02 inch thick was applied on metal the coating would be immersed in water at 185° F. for far in excess of 10 hours without blistering. Gas carbon black is usually very finely divided and 100% will pass a 100-mesh testing sieve.

Summarizing the foregoing, those finely-divided mineral materials which can be preferably used according to this invention so as to afford roofings which are both highly fire resistant and highly blister resistant are finely-divided asbestos-form mineral fibers and finely-divided asbestos-cement scrap or other mixtures of asbestos-form mineral fibers with hydrated Portland cement. Other examples of finely-divided mineral fillers which can be used to afford both fire resistance and blister resistance are hydrated Portland cement and finely-divided serpentine rock. A material which provides high blister resistance but is not effective to afford fire resistance is gas carbon black. If desired, mixtures of the mineral fillers may be employed. If such a mixture is used it is desirable that those materials which have been pointed to as preferable be used as the major proportion of the mineral filler. Moreover, it is preferable that at least about 20% of the filler be fibrous in character, e. g. asbestos-form mineral fiber or asbestos-cement, although when about 20% by weight of the filler is gas carbon black the viscosity of the coating composition is markedly increased due to the great effectiveness of this material in this regard. Preferably the materials having the effective properties above mentioned are used as the entire filler in the special coating composition but minor proportions of other filler materials may be utilized while still retaining some of the advantages of this invention. Of the finely-divided mineral fillers which may be added as diluent filler materials to the highly effective filler materials above described, kaolin clay, calcium silicate hydrate, fly ash, land plaster (uncoarsened gypsum) and fibrous talc are to be preferred. These finely-divided mineral fillers have the property of increasing somewhat the viscosity at 170° F. and resistance to water at 185° F. of bituminous coating compositions as compared with materials such as slate flour or powdered limestone which latter are the filler materials most generally used heretofore in coatings for prepared asphalt roofings and which do not have effectiveness in these respects. Of these materials kaolin clay and calcium silicate hydrate are finely-divided fillers of the flow resistant type that provide a stable and fire resistant skeletal mat when a bituminous composition containing them or either of them is exposed to flame temperature. The substance "fly ash" is the finely-divided coal ash carried to the stack in furnaces burning powdered coal and is collected by electrical precipitation or other suitable method. It usually contains about 95% to 100% of particles

passing a 100-mesh testing sieve. The fibrous talc referred to is exemplified by that fibrous talc which is obtained from deposits in St. Lawrence county, New York, and sold under the name Asbestine. It is used extensively in the paint industry.

The term "mineral" filler is used herein to distinguish from fillers composed of vegetable or animal material and the term "filler" denotes any finely-divided material adapted to be carried in the bitumen matrix of the coating material.

Regardless of the type of filler used it should be of the relatively finely-divided character mentioned above, namely, not more than .0% should be retained on a 10-mesh testing sieve and preferably not more than 10% should be retained on a 14-mesh testing sieve. Moreover, preferably all of the material should pass a 14-mesh testing sieve and not more than 50% should be retained on a 48-mesh testing sieve. This is important both from the point of view of effectiveness in imparting blister resistance and fire resistance and from the point of view of providing a coating that can be spread to uniform thickness and of uniform consistency.

The amount of filler that is desirable depends partly upon the softening point of the bitumen that is used and partly upon whether or not fire resistance is desired in addition to blister resistance. As mentioned above a bitumen having a very high softening point, that is 300° F. or above can be used without any filler as far as blister resistance is concerned but cannot be so used if fire resistance is also desired.

When bitumens having a softening point lower than 300° F. are used, the use of filler becomes important from the standpoint of becoming in order to impart viscosity to the coating composition as a whole. Where a bitumen having a softening point of about 220° to 240° F. is used the presence of about 25% to 55% by weight of filler is usually required. When a bitumen having a softening point below 300° F. is used the filler in amount and type should preferably elevate the softening point of the mixture as a whole to about 300° F. or higher. It is preferable that the special coating having high blister resistance contain at least about 25% by weight of finely-divided filler material of the character herein described. The specific effect of the filler in increasing the softening point and viscosity of the bitumen depends for the most part upon the size and surface characteristics of the filler particles. Preferably the filler should be such that the desired high softening point and high viscosity is attained when using less than about 50% of filler in the bituminous coating composition as a whole.

Excessive amounts of filler tend to make the coating material excessively brittle and low in pliability. In order to preserve desirable pliability for roll roofing and the like, the per cent. elongation at 77° F. of the bituminous material should be at least about 35 and preferably should be at least about 45. The work capacitance of the bituminous material at 77° F. should be at least about 2000 gm.-cm. per cu. cm. and preferably should be at least about 2500 gm.-cm. per cu. cm.

For roofing using a porous sheet material such as felt impregnated with a bituminous saturant and containing about 5% to 10% voids therein as determined by the vacuum amyl alcohol or water absorption test, it is desirable that the

coating be distributed so as to be at least approximately 15 pounds per 100 square feet in order to afford strength adequate for blister resistance in high degree. Where the coating is applied over a non-porous base material such as metal or felt that has been filled and sealed in a substantially completely devolved condition by the use of the method and apparatus of our Patents Nos. 2,105,531 and 2,159,587 the coating may be quite thin, e. g., about 10 pounds per 100 square feet. At the other extreme the blister barrier coating can be applied so as to be very thick and can be applied in one or more layers.

From the standpoint of fire resistance there should be present in the roofing sufficient mineral filler having the surface characteristics above referred to not only to provide a residual skeletal mat which remains in place when the roofing is exposed to flame temperatures, but also to provide a residual skeletal mat that has definite heat insulating capacity. Thus in the foregoing example comprising three thicknesses of preformed shingle material each thickness of the preformed material carried about 50 pounds per 100 square feet of coating material containing at least about 40% of asbestos dust. The coating for each thickness therefore contained about 20 pounds per 100 square ft. of asbestos dust and the roof covering as a whole contained at least about 60 pounds per 100 sq. ft. of roof deck covered. More generally, for fire resistance the coating material should contain at least 30% by weight and preferably at least about 35% by weight of finely-divided heat resistant material having the surface characteristics above mentioned. For high resistance to flame spread at least about 55% by weight of finely-divided heat resistant material should be included in the special coating and preferably at least about 60% by weight of finely-divided heat resistant material should be incorporated.

It is usually preferable that the coating material contain a major proportion of bitumen although the bitumen content may be reduced to about 45% by weight. Conversely, the total amount of filler should not exceed about 55% by weight of the coating composition and preferably is less than 50% by weight. Those compositions which contain about 40% to about 50% by weight of mineral filler are especially fire resistant when the filler has the surface characteristics described herein. Such roofings are especially resistant to bleeding of bitumen from the finely-divided heat resistant material when the coating material is exposed to flame temperatures. Bitumen that bleeds to the surface burns more freely than when mixed with finely-divided heat resistant filler and tends to promote spread of flame. While a small amount of bleeding is not especially harmful, highest resistance to flame spread tests are obtained when the coating material is substantially non-bleeding at flame temperatures. Usually for high fire resistance combined with high blister resistance the coating material is applied to the weather exposed side of the base sheet so that it will weigh about 20 to 70 pounds per 100 sq. ft., although about 30 to 60 pounds per 100 sq. ft. is usually preferable.

In the coating as applied there should, in order to afford a heat insulating mat, be present finely-divided heat resistant material weighing at least about 6 lbs. per 100 sq. ft. and preferably at least about 9 pounds per 100 sq. ft. In the complete roof deck covering as applied compris-

ing one or more layers of the special coating for high fire resistance, the roofing should preferably contain at least about 27 pounds per 100 sq. ft. of finely-divided heat resistant material, and at least 15 pounds per 100 sq. ft. of finely-divided heat resistant material is desirable. As aforesaid, the special coating material is preferably disposed in a plurality of layers separated by felt or other sheet material, each layer however, containing sufficient of the finely-divided heat resistant material to provide a coherent mat of substantial thickness when the coating material is subjected to flame temperatures.

The disposition of the special coating material on the roof covering as a whole may be varied. Thus the special coating may be applied to one side only instead of to both sides of a base sheet. Roof covering material may be disposed in three layers of preformed material as in Figs. 4 to 7, or in two layers as in Figs. 8 and 9, or in a single thickness. The thickness of the coating material in different layers may be varied. For example, one thick layer of special coating may replace two or more thinner layers in a roof deck covering with attainment of comparable results although disposition of the special coating material in a plurality of strata is regarded as preferable from the standpoint of effectiveness in fire resistance inasmuch as the outer strata can then take the brunt of the heat of the flame and the inner strata will protect the roof deck from the highly heated outer strata. Moreover, two like or unlike layers of the special coating material may be contiguous or separated by materials other than sheet materials, e. g., by a layer of mineral granules or the like. Two relatively thin layers of special but unlike coating composition that are contiguous or substantially contiguous may be used to provide what is in effect a single layer of desired thickness and is to be regarded as the equivalent of a single layer. By varying the number, thickness and composition of the layers of special coating composition varying degrees of fire resistance and also blister resistance can be attained in the roofing as a whole. It is preferable, however, to construct roof covering so that they will pass the Underwriters' Laboratories' fire retardant tests. For maximum fire resistance and maximum blister resistance it is preferable that substantially all of the bituminous coating material in prepared roofing materials or in roof coverings as a layer of substantial thickness covering the sheet material be the special coating material herein described. From the point of view of blister resistance the weather exposed coating layer plays the most important role, although, especially in built-up roofings where gases and vapors may be entrapped between layers of sheet material, blistering is likely to occur and can be minimized by the employment of special high viscosity bituminous waterproofing material according to this invention.

When a plurality of coatings is applied in the manufacture of roofings according to this invention the special coating may be combined with other coatings either overlying or underlying the special coating. For example, a sealing coating of bitumen having a softening point of about 240° to 300° F. may be interposed between the special coating and a suitable base sheet or may overlie the special coating. If an overlying coating is substantially water impervious and adapted to seal the special coating from moisture penetration from the weather exposed surface of the

roofing it is not absolutely essential that the material of the coating in itself be resistant for 2 hours or more to water at 185° F. Inasmuch as the special coating is sealed from contact with moisture by the overlying coating so that moisture does not have access to the coating or layer of special composition; and in such case somewhat greater latitude in selection of filler materials from the point of view of preferential affinity for bitumen and water absorption is permitted. When a coating or layer of special coating composition is protected by water impervious or substantially water impervious material overlying the weather exposed surface thereof, the non-water absorptiveness of the special coating or layer as protected by the impervious or substantially impervious material can be tested by applying the special high viscosity coating composition of the thickness in question to a metal panel, e. g. aluminum, and covering it with the water impervious or substantially water impervious material, and if the layer of special high viscosity coating as thus protected does not develop substantial blisters for a period of about 2 hours and preferably for a period of at least about 10 hours, the body portion of the special high viscosity coating is to be regarded as sealed against penetration by water at 185° F. for such periods. When the body portion of the coating or layer of special high viscosity coating material is thus sealed against penetration by water at 185° F. the roofing will be found to be highly resistant to blistering under conditions of weather exposure at normal summer sun roof surface temperatures. The use of a coating composition which in itself is inherently non-water absorptive and resistant to water at 185° F. for such periods is of course preferable, and the body portion of any such coating or layer likewise is regarded as sealed against moisture penetration by water at 185° F.

Other arrangements comprising a plurality of coatings or layers are also possible in the practice of this invention.

The base material on which the special coating is used is optional, although for economy of manufacture bituminized felt is desirable. The felt generally weighs about 3 to 15 pounds per 100 sq. ft. In fact it is one of the advantages of the present invention that asphalt roofing comprising ordinary bituminized roofing felt as a base which is at best somewhat water absorptive because of the occurrence therein of vegetable fibers can be made very highly resistant to blistering and also in preferred embodiments very fire resistant. Any suitable fiber such as cotton, jute, cellulose, wool, wood and the like may be used. If the felt is composed wholly or partially of mineral fiber further increased fire resistance can be afforded. In addition to felt other sheet or web materials, fibrous or non-fibrous and foraminous or non-foraminous, such as fibrous sheets woven or otherwise, paper or the like may be used. Moreover, metal protected from the weather by the high viscosity bituminous coating may be used as a roofing according to this invention. When it is said that the base sheet material is coated or adherently coated with a coating or layer of special bituminous coating composition, the coating or layer may or may not be directly in contact with the base sheet.

While the high viscosity bituminous material is ordinarily applied as a coating or layer or less impregnation in the base material, sometimes occur especially when the bituminous material contains a very finely-divided filler such

as gas carbon black which can readily penetrate the base material or when no filler whatever is employed. For this reason the blister barrier material may occur either as a coating layer or as an impregnating layer in roofings embodying this invention. When the base sheet is coarsely interstitious the special coating composition, even when containing a fibrous filler, will tend to enter substantially into the body portion of the base sheet.

The special coating material may be used with or without a surface application such as slate granules although ordinarily this or some other material, preferably mineral, is caused to adhere to the surface, e. g., mica flakes, pulverized talc, etc. It is an advantage of the present invention that any granular material that is caused to adhere to or be embedded in the special coating is highly resistant to removal by impact or abrasive influences both at summer sun temperatures and at lower temperatures.

The back or non-weather exposed side of the roofing, when the roofing comprises a bituminized fibrous base sheet, is usually covered with some finely-divided material such as mica flakes. If desired cork granules may be partially embedded in a bituminous coating, which may or may not be mineral filled, on the back of the roofing. The cork granules serve to increase somewhat the thermal insulating effect of the roofing and assist in protecting an underlying combustible roof deck when the roofing is subjected to flame as by a burning brand in contact with the weather surface of the roofing.

From the foregoing it is apparent that according to the present invention a roofing is afforded which greatly improves upon bituminous roofings heretofore made. Various improvements above described are preferably all availed of in the practice of this invention although part only of the improvements may be utilized and still attain some of the advantages of this invention. As mentioned above a most important attribute of the new roofing is its very high resistance to blistering which sets it apart from any roofing known to us that has been made heretofore. That roofing which are blister resistant can be made which are likewise highly fire resistant is also a most important feature of this invention.

While this invention has been described in connection with certain specific embodiments thereof it is to be understood that this has been done for illustrative purposes only and that the practice of this invention may be varied within the scope of the language of the following claims considered in the light of the foregoing description of our invention.

#### We claim:

1. A roofing comprising a blister barrier waterproofing layer of thermoplastic bituminous material which is carried by sheet-like base material and which is of substantially uniform composition throughout and is of a thickness not less than that corresponding to about 10 pounds per 100 square feet of the roofing throughout the extent of said layer where said layer is designed to provide blister resistance, the thermoplastic bituminous material of said layer containing at least about 45% by weight of bitumen, and said thermoplastic bituminous material having a softening point above 300° F., having a viscosity of at least about  $4 \times 10^4$  poises at 170° F., having a tensile strength of at least about 150 grams per square centimeter at 170° F., having a percent elongation at 77° F. of at least about 35, and

having a work capacitance at 77° F. of at least 2,000 gm. cm. per cu. cm., and said thermoplastic bituminous material when heat-plasticized being spreadable to form a layer of uniform consistency and uniform thickness and said bituminous waterproofing layer being uniformly throughout resistant for at least about two hours to water at 185° F.

2. A roofing according to claim 1 wherein said blister barrier waterproofing layer of thermoplastic bituminous material is applied to the weather side of a fibrous web base sheet containing less than 2% of voids.

3. As an article of manufacture, bituminized roofing comprising a bitumen impregnated fibrous web having overlying one side thereof a blister barrier waterproofing layer of thermoplastic bituminous material which layer is of substantially uniform composition throughout and of uniform thickness that corresponds to not less than 10 pounds per 100 square feet, the thermoplastic bituminous material of said layer containing at least about 45% by weight of asphaltic bitumen, and said thermoplastic bituminous material having a softening point above 300° F., but not substantially above about 350° F., having a viscosity at 170° F. in excess of  $20 \times 10^4$  poises, having a tensile strength at 170° F. of at least 150 gms. per sq. cm., having a percent elongation at 77° F. above about 45, and having a work capacitance at 77° F. of at least about 2,000 gm. cm. per cu. cm., and said thermoplastic bituminous material when heat-plasticized being spreadable to form a layer of uniform consistency and uniform thickness, and said bituminous waterproofing layer being uniformly throughout resistant for at least 10 hours to water at 185° F.

4. A roofing comprising a blister barrier waterproofing layer of bituminous coating composition which is carried by sheet-like base material and which contains at least about 45% to about 70% by weight of bitumen having a softening point above about 200° F. and not above approximately 240° F. together with about 30% to about 65% by weight of water-insoluble finely-divided solid non-porous mineral filler, at least 30% by weight of said coating composition consisting of said water-insoluble finely-divided solid non-porous mineral filler which has a preferential affinity for said bitumen as against water at 185° F., said layer of said coating composition being of a thickness not less than that corresponding to about 10 pounds per 100 square feet throughout the extent of said layer where said layer is designed to provide blister resistance, said coating composition having a softening point above 300° F., having a viscosity at 170° F. of at least about  $4 \times 10^4$  poises, having a per cent. elongation at 77° F. of at least 45, having a tensile strength at 170° F. of at least 150 gms. per sq. cm., and having a work capacitance at 77° F. of at least about 2,000 gm. cm. per cu. cm., and said coating composition when heat-plasticized being spreadable to form a layer of uniform consistency and uniform thickness.

5. A roofing according to claim 4 wherein said sheet-like base material is a fibrous web substantially completely filled with a bituminous saturant and sealed on the opposite surfaces thereof in a substantially devolved condition with sealing layers of bituminous coating material integrally bonded with the saturant and with the fibers of the web adjacent the surfaces, said sealing layers filling the surface interstices of the web to substantial depth with avoidance of sub-

stantial porosity and wherein said fibrous web has on the weather side thereof said blister barrier waterproofing layer of thermoplastic bituminous composition.

6. A roofing comprising a blister barrier waterproofing layer of thermoplastic bituminous coating composition which is carried by sheet-like base material, said thermoplastic bituminous composition containing at least about 45% by weight of bitumen having a softening point between about 200° F. and about 275° F. and uniformly distributed therein water-insoluble finely-divided solid mineral filler material having preferential affinity for said bitumen as against water at 185° F., said bitumen with said filler material having a preferential affinity for said bitumen as against water at 185° F. distributed therein having a softening point above 300° F., said coating composition having a viscosity at 170° F. greater than  $4 \times 10^4$  poises, having a tensile strength at 170° F. of at least 150 gms. per sq. cm., having a per cent. elongation at 77° F. of at least 35, and having a work capacitance at 77° F. of at least 2000 gm. cm. per cu. cm., and said coating composition when heat-plasticized being spreadable to form a layer of uniform consistency and uniform thickness, and said layer of said coating composition being of a thickness throughout not less than that corresponding to about 10 pounds per 100 square feet throughout the extent of said layer where said layer is designed to provide blister resistance.

7. A roof covering comprising at least one water-proofing blister barrier layer of highly fire-resistant thermoplastic bituminous coating material, and sheet material which carries said blister barrier waterproofing layer as a coating, said thermoplastic bituminous coating material containing about 45% to about 70% by weight of bitumen having a softening point between about 200° F. and about 300° F. and dispersed in and mixed with said bitumen about 30% to about 55% by weight of finely-divided water-insoluble solid heat resistant mineral filler, at least 30% by weight of said coating material consisting of said finely-divided water-insoluble solid heat resistant mineral filler which passes a 14 mesh testing sieve and over 50% by weight of which is retained on a 200-mesh testing sieve, which has a preferential affinity for said bitumen as against water at 185° F. which constitutes at least about 15 pounds per 100 square feet of said roof covering, and which has a flow resistance value of at least 40 minutes when used in a bituminous composition within the range consisting of 30% to 55% of said filler and 45% to 70% of bitumen having a softening point of 200° F. to 300° F. that is subjected to flame temperature under the flow resistance test as defined herein, said layer being of substantially uniform composition throughout and of a thickness not less than corresponding to about 10 pounds per 100 square feet of the roofing throughout the extent of said layer where said layer is designed to provide blister resistance, and said thermoplastic bituminous coating material having a softening point above 300° F., having a viscosity at about 170° F. of at least about  $4 \times 10^4$  poises, having a tensile strength at 170° F. of at least 150 gms. per sq. cm., having a percent elongation above about 35 at 77° F. and when heat-plasticized being spreadable to form a layer of uniform consistency and uniform thickness.

8. A roof covering according to claim 7, wherein said roof covering includes a plurality of

sheets of sheet material and a plurality of layers of said thermo-plastic bituminous coating material, one of said layers of coating material covering the outermost of said sheets of sheet material and another layer of said thermoplastic bituminous coating material interposed between said sheets of sheet material, each of at least two layers of said coating material containing at least 6 pounds per 100 square feet of said finely-divided water-insoluble solid heat resistant material which passes a 14-mesh testing sieve and over 50% by weight of which is retained on a 200-mesh testing sieve, which has a preferential affinity for said bitumen as against water at 185° F., which constitutes at least 15 pounds per 100 square feet of said roof covering and which has a flow resistance value of at least 40 minutes when used in a bituminous composition within the range consisting of 30% to 55% of said filler and 45% to 75% of bitumen having a softening point of 200° F. to 300° F.

9. A roof covering according to claim 7, wherein said roof covering includes a plurality of sheets of said sheet material and a plurality of layers of said thermoplastic bituminous coating material and wherein said layers of sheet material and said layers of thermoplastic bituminous coating material are bonded together in the form of a built-up roofing.

10. A prepared roofing material comprising a waterproofing blister barrier layer of highly fire-resistant bituminous coating material and sheet material which carries said waterproofing layer as a coating, said coating material containing asphaltic bitumen having a softening point between about 200° F. and about 240° F. of the range 50% to 60% by weight of the coating material and dispersed in and mixed uniformly with said bitumen finely-divided solid water-insoluble heat-resistant mineral filler, at least 40% by weight of said coating material consisting of said finely-divided water-insoluble solid heat-resistant mineral filler which passes a 14-mesh testing sieve and over 50% by weight of which is retained on a 200-mesh testing sieve, which has a preferential affinity for said bitumen as against water at 185° F., and which has a flow resistance value of at least 40 minutes when used in a bituminous composition within the range consisting of 40% to 50% of said filler and 50% to 60% of bitumen having a softening point of 200° F. to 300° F. that is subjected to flame temperature under the flow resistance test as defined herein, said layer being of substantially uniform composition throughout and of a thickness not less than that corresponding to about 20 pounds per 100 square feet of the roofing throughout the extent of said layer where said layer is designed to provide blister resistance, and said coating material having a softening point above 300° F., having a viscosity at 170° F. of at least  $20 \times 10^4$  poises, having a tensile strength at 170° F. of at least 150 gms. per sq. cm., having a work capacitance at 77° F. of at least 2,000 gm. cm. per cu. cm., the filler content of said coating material not containing more than about 10% by weight of particles retained on a 14-mesh testing sieve, and said roofing material being pliable at 77° F.

11. A roof covering in the form of a built-up roofing which comprises a plurality of sheets of fibrous sheet material impregnated with an asphaltic saturant and bonded in integral relation and interposed between at least two of said sheets, a blister barrier layer of waterproofing bituminous material, said bituminous water-

proofing material containing at least about 45% by weight of asphaltic bitumen, and said bituminous waterproofing material having a viscosity at 170° F. of at least  $4 \times 10^5$  poises, having a softening point above 300° F., having a per cent. elongation at 77° F. of at least 35, and having a work capacitance at 77° F. of at least 2,000 gm. cm. per cu. cm., and said bituminous waterproof material when heat-plasticized being spreadable to form a layer of substantially uniform consistency and uniform thickness.

12. A roof covering in the form of a built-up roofing according to claim 11 wherein at least one of said layers is a blister barrier bituminous coating maintained in bonded relation with a sheet of said fibrous sheet material by a bituminous adhesive consisting essentially of bitumen having a softening point above 300° F.

13. A roofing comprising a blister barrier waterproofing layer of thermoplastic bituminous composition that contains at least about 45% to about 70% by weight of bitumen having a softening point between about 200° F. and about 300° F. and substantially uniformly dispersed in and mixed with said bitumen as a filler about 30% to about 55% by weight of finely-divided filler material selected from the following groups, a major proportion of said filler being selected from the group consisting of asbestos-form mineral fiber which passes a 10 mesh testing sieve and over 50% by weight of which is retained on a 200 mesh testing sieve, asbestos-cement of similar particle-size, hydrated Portland cement passing a 14 mesh testing sieve, serpentine rock passing a 14 mesh testing sieve, gas carbon black and mixtures of the said materials in said group, and a minor proportion of said filler being selected from the group consisting of kaolin clay, calcium silicate hydrate, fly ash, land plaster, fibrous talc and mixtures of any of said materials in said last mentioned group, said waterproofing layer being of a thickness not less than corresponding to at least about 10 pounds per 100 square feet throughout the extent of said layer designed to provide blister resistance and said thermoplastic bituminous composition having a softening point above 300° F., having a viscosity at 170° F. of at least  $4 \times 10^5$  poises, having a per cent elongation of at least 35 at 77° F., and when heat-plasti-

cized being spreadable to form a layer of uniform consistency and uniform thickness.

14. A roofing comprising a blister barrier waterproofing layer of thermoplastic bituminous composition that contains about 45% to about 70% by weight of bitumen having a softening point between about 200° F. and about 300° F. and substantially uniformly dispersed in and mixed with said bitumen as a filler about 30% to about 55% by weight of finely-divided filler material selected from the group consisting of asbestos-form mineral fiber which passes a 10 mesh testing sieve and over 50% by weight of which is retained on a 200 mesh testing sieve, asbestos-cement of similar particle size, hydrated Portland cement passing a 14-mesh testing sieve, serpentine rock passing a 14-mesh testing sieve, gas carbon black and mixtures of any of the said materials in said group with the proviso that said thermoplastic bituminous composition shall contain gas carbon black, said waterproofing layer being of a thickness not less than that corresponding to at least about 10 pounds per 100 square feet throughout the extent of said layer where said layer is designed to provide blister resistance and said thermoplastic bituminous composition having a softening point above 300° F., having a viscosity at 170° F. of at least  $4 \times 10^5$  poises, having a per cent. elongation of at least 35 at 77° F., and when heat-plasticized being spreadable to form a layer of uniform consistency and uniform thickness.

15. A roofing having a blister barrier waterproofing layer of thermoplastic bituminous composition carried by sheet material, said layer containing at least about 45% by weight of bitumen having a softening point between about 200° F. and about 300° F. and at least about 25% by weight of gas carbon black, said composition having a softening point above 300° F., having a viscosity of at least  $4 \times 10^5$  poises at 170° F., having a per cent. elongation at 77° F. of at least 35, and when heat-plasticized being spreadable to form a layer of uniform thickness and uniform consistency, and said layer being of a thickness not less than corresponding to about 10 pounds per 100 square feet throughout the extent of said layer designed to provide blister resistance.

GEORGE ARTHUR FASOLD.  
HAROLD W. GREIDER.

## CERTIFICATE OF CORRECTION.

Patent No. 2,326,724.

August 10, 1943.

GEORGE ARTHUR FASOLD, ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 3, first column, line 54, for "dusing" read --dusting--; page 4, first column, line 59, for "viscosity" read --viscosity--; page 5, first column, line 33, for "chrysolite" read --chrysotile--; page 8, first column, line 16, for "skeleton" read --skeletal--; and second column, line 55, for "bituminous" read --bituminized--; page 9, first column, line 4, for "with" read --without--; line 30, for "build-up" read --built-up--; line 40, for "rust" read --dust--; page 15, second column, line 19, for "abestiform" read --asbestiform--; page 20, first column, line 26, claim 6, for "plasticised" read --plasticized--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 12th day of October, A. D. 1943.

(Seal)

Henry Van Arsedale,  
Acting Commissioner of Patents.

**Exhibit C – Wikipedia Entry on “Basal Cleavage” Printed on  
January 7, 2007**



## Cleavage (crystal)

From Wikipedia, the free encyclopedia  
(Redirected from Basal cleavage)

**Cleavage**, in mineralogy, is the tendency of crystalline materials to split along definite planes, creating smooth surfaces, of which there are several named types:

- **Basal cleavage:** cleavage parallel to the base of a crystal, or to the plane of the lateral axes. This occurs quite easily in graphite, making the material feel slippery.
- **Cubic cleavage:** cleavage parallel to the faces of a cube. This is the source of the cubic shape seen in crystals of ground table salt (sodium chloride).
- **Diagonal cleavage:** cleavage parallel to a diagonal plane.
- **Lateral cleavage:** cleavage parallel to the lateral planes.
- **Octahedral, dodecahedral, or Rhombohedral cleavage:** cleavage parallel to the faces of an octahedron, dodecahedron, or rhombohedron (respectively). Octahedral cleavage is seen in common semiconductors (see below)
- **Prismatic cleavage,** cleavage parallel to a vertical Prism.



This is of technical importance in the electronics industry and in the cutting of gemstones. While precious stones are generally cleaved by impact, man-made single crystals of semiconductor materials are generally sold as thin wafers which are much easier to cleave. Simply pressing a silicon wafer against a soft surface and scratching its edge with a diamond scribe is usually enough to cause cleavage; however, when dicing a wafer to form chips, a procedure of scoring and breaking is often followed for greater control. The vast majority of commercial semiconductors (Si, Ge, GaAs, InSb, etc.) are diamond cubic, a space group for which octahedral cleavage is observed. This means that some orientations of wafer allow near-perfect rectangles to be cleaved.

### See also

- Crystal structure
- Facet
- Fracture

### References

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Clcavage (crystal) - Wikipedia, the free encyclopedia

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Categories: Crystallography | Mineralogy

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